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**JET PROPULSION LABORATORY
CALIFORNIA INSTITUTE OF TECHNOLOGY
PASADENA, CALIFORNIA**

(NASA-CR-51289) SPHERICAL HIGH-ENERGY
SOLID-PROPELLANT ROCKET MOTOR (Atlantic
Research Corp.) 214 p

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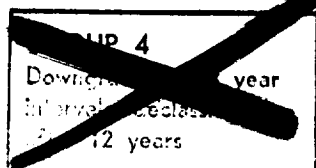
**FINAL REPORT ON THE SPHERICAL HIGH-ENERGY
SOLID-PROPELLANT ROCKET MOTOR (U)**

For
**JET PROPULSION LABORATORY
CALIFORNIA INSTITUTE OF TECHNOLOGY
Pasadena, California**

**Available to U.S. Government Agencies and
U.S. Government Contractors Only.**

**JPL Contract 950097
Report Number ARC-SR-23A1
March 1, 1963**

By
**ATLANTIC RESEARCH CORPORATION
Alexandria, Virginia**



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ARC-SR-23A1

This report covers the work accomplished by Atlantic Research Corporation in the development of a high-performance solid-propellant rocket motor for the Jet Propulsion Laboratory of the California Institute of Technology. The program, authorized under JPL Contract No. 950097, was initiated on 3 August 1961 and terminated on 22 August 1962.

The report has been reviewed for technical accuracy and released by the undersigned.

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SUMMARY

A design and development program was conducted in an attempt to advance the state-of-the-art of solid-propellant rocket motor technology beyond its status at the time of contract in August 1961. The primary program objective was the design of a 36-inch-diameter spherical motor having a higher propellant specific impulse and a higher propellant mass fraction than that of any existing rocket motor. The prototype motor design was to incorporate a propellant formulation and component design concepts which had been successfully tested during the course of the program. Final static firing evaluation was to be conducted in 17-inch-diameter subscale motors.

The use of hydrazine nitroform (HNF) in a beryllium-containing, plasticized nitrocellulose binder was investigated as a means of meeting the requirement for a propellant significantly higher in performance than available state-of-the-art propellants. The thermodynamic specific-impulse values at standard conditions of such formulations are in the vicinity of 288 to 290 lb-sec/lb.

The chemistry of HNF with respect to other propellant ingredients was intensively studied. It was found that the presence of ferric oxide and moisture as impurities in HNF results in a catalytic oxidation in which both NO and N_2O are evolved. Elimination of the impurities greatly improves thermal stability. The presence of trace quantities of moisture in propellant formulation results in hydrolysis of ester-type plasticizers (e.g., triacetin), which, in turn, results in degradation of HNF. It was therefore necessary to eliminate or reduce the concentration of such plasticizers in the formulation. The stability of trimethylolethane trinitrate (TMETN)-plasticized formulations was satisfactory.

It was found that the as-received needle-shaped HNF could be ultrasonically recrystallized to obtain symmetrical particles. This advance resulted in higher solids loadings, improved processing characteristics, lower burning rate, and lower pressure exponent.

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Burning rates and pressure exponents were measured in a strand burner for many formulations. The design of the 17-inch subscale motor required a burning rate of 0.23 in/sec and a pressure exponent less than 0.5 at a pressure of 700 psia. These parameters were achieved in formulations containing ester-type plasticizers (which had to be rejected because of stability considerations), but could not be achieved in the stable TMETN-plasticized formulations.

In an effort to obtain a propellant which would have satisfactory burning rates and pressure exponents for the 17-inch motor design, attention was directed to a TMETN-plasticized polyurethane binder containing beryllium and HNF. The thermodynamic specific impulse values at standard conditions of such formulations are in the vicinity of 293 lb-sec/lb. In a JPL X-535 polyurethane binder, the polypropylene glycol dissolves some of the HNF which in turn reacts with toluene diisocyanate (TDI). To overcome this obstacle, a non-glycol prepolymer based on castor oil and TDI was used. To improve processability as well as performance, TMETN was added to the system. Two test motors were satisfactorily loaded with the resulting formulation, but both motors failed in static firing because of an excessively high pressure exponent. Replacing half of the HNF with ammonium perchlorate reduced the pressure exponent to 0.55 and the thermodynamic specific impulse at standard conditions to 287 lb-sec/lb. Two small test motors containing the aluminum analog of this propellant were satisfactorily fabricated and fired. This promising work had to be discontinued because of time and budgetary limitations.

A beryllium-containing polyurethane propellant oxidized with ammonium perchlorate was tailored for use in testing of the 17-inch subscale motor. The theoretical specific impulse values at standard conditions of such formulations are in the vicinity of 283 lb-sec/lb.

Problems were encountered in obtaining reproducible cures with the beryllium-polyurethane propellants. The cause of these difficulties was found to be associated with the beryllium powder. The curing problem was resolved by the judicious specification of the beryllium powder and by formulating and processing techniques.

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A small-scale static firing program was conducted to optimize the propellant performance. Forty-two firings were conducted at both sea-level and simulated-altitude conditions in standard Rohm and Haas 6C11.4 test motors containing a nominal 10 pounds of propellant. Specific impulse efficiencies obtained from the simulated-altitude firings were found to be significantly lower than those obtained from sea-level firings. These differences in efficiencies were attributed to losses from super-cooling of the metal oxide during the expansion process at the higher nozzle expansion ratios utilized in the simulated altitude firings. One of the propellants investigated in this program, Arcane 38, was static-fired at a higher thrust level in a subsequent program (Contract AF 04(611)-8180) giving a delivered specific impulse (at standard conditions and a 15-degree cone angle) of 260 lb-sec/lb.

A spherical grain was designed for the 36-inch-diameter motor to afford the maximum attainable loading density compatible with a nearly constant burning surface. Scaling of grain dimensions for the 17-inch motor gave a web of 5.11 inches and a burning time of 21.7 seconds. Minor dimensional adjustments were subsequently effected to counteract regressive burning characteristics exhibited in the subscale motor firings.

Two types of 17-inch-diameter subscale motors were designed for static firing tests: a heavy-walled steel motor and a titanium motor of lightweight wall thickness. The steel case was fabricated from AISI 4130 steel and had a wall thickness of 0.075 inch; the titanium case consisted of 6AL-4V titanium alloy and had a wall thickness of 0.030 inch. Six steel and seven titanium cases were fabricated. All of the heavy-walled steel motors were statically fired; the firing of titanium units was prohibited by the depletion of contractual fund allocations. The titanium case was determined to be of adequate strength in a hydrostatic pressure test of 1,150 psig, conducted at the Wyle Laboratories, El Segundo, California. Based on the results of this test, a minimum-weight, 36-inch-diameter titanium spherical motor case was designed in accordance with the stress criteria specified by the Jet Propulsion Laboratory.

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An asbestos-phenolic nozzle with a graphite nozzle-throat insert was designed to be completely submerged within the subscale motor case. In the initial design, the nozzle was to be retained by attachments fastened to the motor case by means of the studs in the case flange. Subsequent tensile failures in the 17-inch motor firing program and in hydrostatic pressure tests, however, showed this design to be unsuitable. The subscale nozzle was therefore redesigned to incorporate a heavy steel retaining ring in place of the integral flange on the asbestos-phenolic cone. The nozzle for the full-scale, 36-inch spherical motor was designed to have an optimum contour and to include a diffuser section affording a greater nozzle expansion ratio. After the redesign of the subscale nozzle, the nozzle for the 36-inch motor was modified to incorporate a titanium retention flange as part of the diffuser section rather than as an integral part of the submerged nozzle cone.

The igniter designed for test in the subscale motor firings was toroidal in shape and constructed from a nylon tube containing two U.S. Flare 908B squibs and 10 to 15 grams of U.S. Flare 2D ignition pellets. This ignition system, however, gave excessive ignition delays. The development of an integral nozzle closure and igniter to correct this problem was initiated but not completed because of the depletion of funds.

Of the six heavy-walled subscale motors fired during the program, three were loaded with beryllium-containing propellants of the Arcane 40 series and three with Arcane 42 propellant, the aluminum analog of Arcane 40. Two of the analog propellant firings were successful; one motor burned for 22 seconds and the other for 18.7 seconds. Three of the four remaining motors failed because of nozzle expulsion problems; the fourth malfunctioned on ignition as the result of an explosive atmosphere created by a 20-second hang-fire situation.

The propellant mass fraction of the final prototype 36-inch spherical motor assembly design was calculated to be 0.922. A weight reduction in nozzle components was considered to be a feasible approach to achieving a higher ratio.

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INTRODUCTION

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This report covers the work accomplished by the Atlantic Research Corporation in the development of an advanced state-of-the-art high-performance solid-propellant rocket motor for the Jet Propulsion Laboratory of the California Institute of Technology. The general approach was to develop a high-performance propellant formulation and to demonstrate the motor design in a series of 17-inch-diameter subscale motor firings. The program, authorized under JPL contract 950097, was initiated on the 3rd of August, 1961 and was terminated on the 22nd of August 1962.

The major portion of the effort was devoted to the investigation of a high-energy hydrazine nitroform (HNF)-beryllium propellant system. Major problems were encountered with this system which required an extensive laboratory effort in an attempt to establish its feasibility. Late in the program, after it was established that the HNF-beryllium system was not feasible for this motor application, the propellant development effort was directed to the tailoring of a polyurethane-ammonium perchlorate-beryllium system. This propellant, Arcane 40, was characterized and its performance determined by firing 10 pound motors in the Atlantic Research simulated altitude facility. As a result of the extended difficulties encountered during the propellant development phase of the program only a small portion of the total effort was devoted towards hardware development. A grain design affording a level pressure-time characteristic was developed, and a 17-inch 6AL-4V titanium case was designed and tested hydrostatically.

Although the initial program objective of demonstrating a high-performance motor was not accomplished, some significant contributions were made in propellant technology. *long.*

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I. DEVELOPMENT AND EVALUATION OF A HYDRAZINE NITROFORM PROPELLANT

INTRODUCTION AND SUMMARY

Hydrazine nitroform (HNF) was used in an effort to develop a high-specific-impulse propellant meeting contractual performance requirements. Theoretical calculations indicated that the use of HNF in either double-base, single-base, or polyurethane-type binders with beryllium as the fuel would result in specific impulses in the region of 290 lb-sec/lb under standard conditions. In late 1961, however, it became evident that propellant development work on HNF was not proceeding as anticipated. The motor design required a propellant with a low pressure exponent and burning rate, but it was found that these could not be achieved. At a conference at the Jet Propulsion Laboratory in November 1961, it was mutually agreed to concentrate the motor effort on polyurethane propellants while still attempting to bring the HNF propellant to a more fully developed state.

This section summarizes the state-of-the-art of hydrazine nitroform and propellants incorporating hydrazine nitroform (HNF) as a result of work done under this contract. In this effort, the chemistry of hydrazine nitroform was studied with respect to nitrocellulose plasticizers, the components of polyurethane binders, and the effects of ferric oxide and moisture impurities. The as-received hydrazine nitroform was ultrasonically recrystallized from particles with a length-to-diameter ratio of 10:1 to particles with a length-to-diameter ratio of 1:1. The more symmetrical particles facilitated propellant processing, solids loading, and improved strand ballistics. The sea-level and altitude theoretical specific impulse and theoretical flame temperature were calculated for a number of systems in which hydrazine nitroform was the oxidizer and beryllium was the fuel, and hydrazine nitroform was successfully incorporated into a double-base formulation plasticized with trimethylolethane trinitrate (TMETN), a modified polyurethane formulation, and a TMETN-plasticized polyurethane formulation. Two 1/4-pound motors were fired using the TMETN-plasticized polyurethane formulation.

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THE CHEMISTRY OF HYDRAZINE NITROFORM

The reaction of nitroform with bases has been studied in detail by Hercules Powder Company¹ and U. S. Rubber Company². Hercules described a synthesis of hydrazine dinitroform, but further investigation by the Naval Propellant Plant³ showed the product of this reaction had properties which more closely resembled tetrazines than hydrazine nitroform. Johnson and Taylor of the Naval Ordnance Laboratory⁴ reported a synthesis of the mono-nitroform salt and described some of its properties. At that time, however, the compound was considered too sensitive for further development. No further work was done until 1960, when Lovett and Brown of Esso Research and Engineering Company⁵ reported that the compound had sufficient thermal stability for propellant applications and that calculations showed the substitution of hydrazine nitroform (HNF) for ammonium perchlorate increased specific impulse by 8 to 12 seconds. On the basis of this information, the Bureau of Naval Weapons requested the Naval Propellant Plant to consider this compound as an oxidizer for a new high-energy propellant.

¹Hercules Powder Company, Monthly Progress Report 12. D. L. Kouba, et al., NOrd 9925, July 1, 1949. ~~CONFIDENTIAL~~

²U. S. Rubber Company, Incorporated, Synthesis of New Explosives and Propellants. NOrd 10129, October 1, 1948 - February 9, 1949.

³U. S. Naval Propellant Plant, R-8 Monthly Progress Report. October - December 1960. ~~CONFIDENTIAL~~

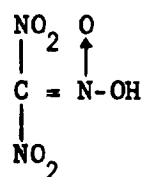
⁴Naval Ordnance Laboratory, Guanidine Nitroformate and Hydrazine Nitroformate as Possible New High Explosives. NavOrd 2125, July 11, 1951. ~~CONFIDENTIAL~~

⁵Esso Research and Engineering Company, Quarterly Progress Report on Research on Advanced Solid Propellants. Report 170. 60 - 2, March 11 - July 10, 1960. ~~CONFIDENTIAL~~

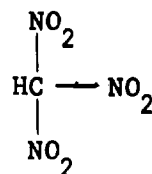
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Beilstein¹ reports two forms of nitroform:



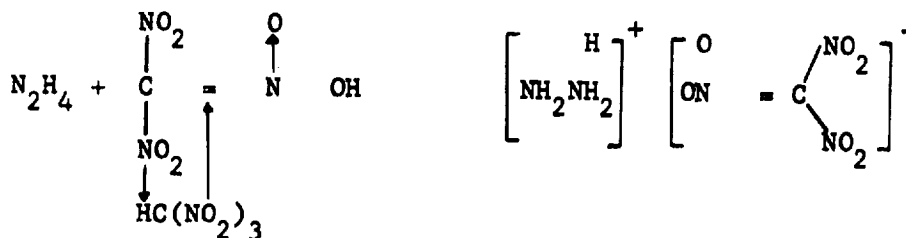
aci-form
m.p. 50°C



trinitromethane
m.p. 16°C

The aci-form is the type of enolization that occurs with most nitro compounds, but in the case of nitroform, this structure is apparently more stable.

The reaction of nitroform and hydrazine to form hydrazine nitroform is a neutralization reaction.



At the Naval Propellant Plant² the reaction is carried out in a methanol solution, after which the crude HNF is precipitated from methanol by carbon tetrachloride, filtered, redissolved in methanol, and recrystallized. The pH of the resulting HNF and its environment is extremely critical. If the pH value is appreciably greater than 5.0 or less than 4.5, either hydrazine or nitroform is liberated. In the presence of moisture, hydrolysis will liberate active hydrazine and nitroform which may react or decompose to form gaseous products when in contact with other propellant ingredients.

¹Beilstein, Friedrich Konrad, Beilsteins Handbuch Der Organischen Chemie. Vol. I, 4th Edition. Berlin: Springer-Verlag, 1948, p. 116.

²U. S. Naval Propellant Plant, TMR-188, Hydrazine Nitroform, Its Synthesis and Properties. W. Blankenship, et al., June 1, 1961. CONFIDENTIAL

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As a result of an apparent stability problem in propellant systems incorporating HNF as the oxidizer, an extensive investigation of the chemical reactions of HNF was conducted. The general chemical reactions of HNF are summarized in Table I.

Several things are immediately apparent from Table I. The normal (nitroglycerin or trimethylolethane trinitrate plasticized) double-base systems seem to be the only inherently stable systems for HNF, provided there is no moisture or other impurities present. However, both moisture and a ferric oxide impurity were found to be present.

The mechanism of decomposition in the above case was the hydrolysis of the HNF by the moisture present into hydrazine and nitroform, and then the reduction of the Fe_2O_3 by the hydrazine to Fe and FeO with the corresponding oxidation of the Fe and FeO by the nitroform back to Fe_2O_3 . The products of decomposition yielded both N_2O and NO as analyzed by mass spectrometer.

Since the as-received HNF is dissolved in methanol and ultrasonically recrystallized before use, a simple filtration of the HNF-saturated methanol solution prior to recrystallization proved to be adequate for removal of the impurity. The HNF manufacturers now are filtering their material prior to crystallization.

It also appears that water hydrolyzes most ester-type nitrocellulose plasticizers. The resultant hydrolyzed ester products react with HNF and produce gassing over long periods of time, depending on the particular ester used.

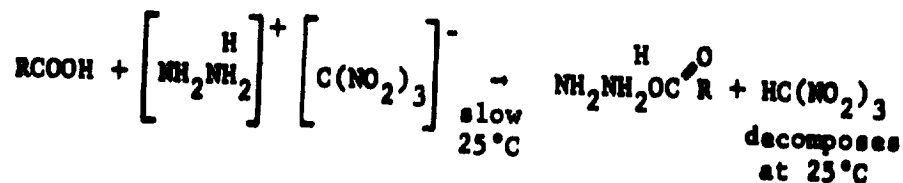
Another reaction of particular interest is the condensation reaction¹ between isocyanates and HNF. This highly exothermic reaction will not take place unless there is a third agent present in solution that will first

¹U. S. Naval Propellant Plant, State-of-the-Art Review of Propellant Formulations Containing Hydrazine Nitroform. JANAF-ARPA-NASA Solid Propellant Group, Bulletin of 18th Meeting, G. A. Kalvin, et al., Volume II, June 1962. CONFIDENTIAL

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TABLE I

1. Organic and Inorganic Acids


$$\text{R}_1\text{R}_2\text{NH} + \left[\begin{array}{c} \text{H} \\ \text{NH}_2\text{NH}_2 \\ \text{HMF} \end{array} \right]^+ \left[\text{C}(\text{NO}_2)_3 \right]^- \xrightarrow{40^\circ\text{C}} \text{R}_1\text{R}_2\text{NHC}(\text{NO}_2)_3 + \text{NH}_2\text{NH}_2$$

amines hydrazine

$$\text{C}_6\text{H}_6 + \text{C} \begin{matrix} \text{O} \\ \text{O} \\ \text{O} \end{matrix} + \left[\text{NH}_2\text{NH}_2 \right]^+ \left[\text{C}(\text{NO}_2)_3 \right]^- \xrightarrow{60^\circ\text{C}} \text{HC}(\text{NO}_2)_3 + \text{decomposition products}$$

$$\text{H}^+ + \text{C}(\text{NO}_2)_3^-$$
$$\text{NH}_2\text{NH}_2 + \left[\text{NH}_2\overset{\text{H}}{\text{NH}_2} \right]^+ \left[\text{C}(\text{NO}_2)_3 \right]^- \xrightarrow{40 \text{ to } 100^\circ\text{C}} \text{combustion products}$$

fuel oxidizer

$$\left[\begin{array}{c} \text{H} \\ \text{NH}_2\text{NH}_2 \end{array} \right]^+ \left[\text{C}(\text{NO}_2)_3 \right]^- \xrightleftharpoons[25^\circ\text{C}]{\text{HOH}} \text{NH}_2\text{NH}_2 + \text{HC}(\text{NO}_2)_3$$

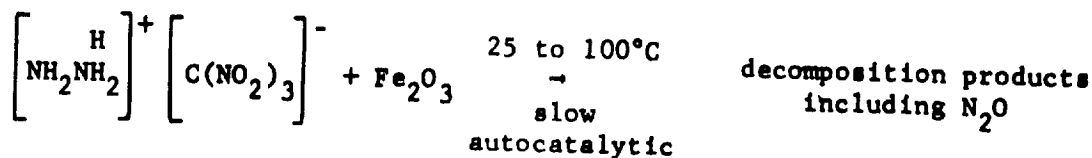
$$\downarrow \uparrow$$

$$\text{H}^+ + \text{C}(\overline{\text{NO}}_2)_3$$

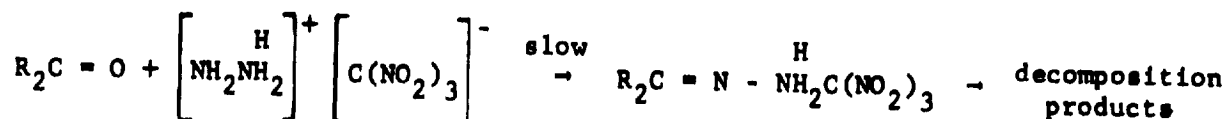
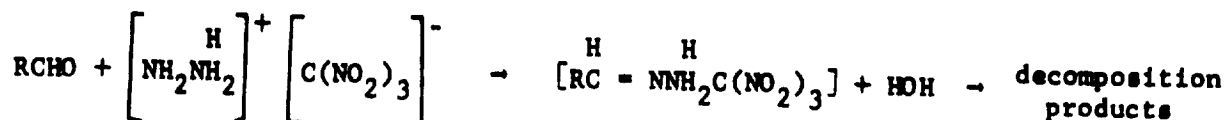
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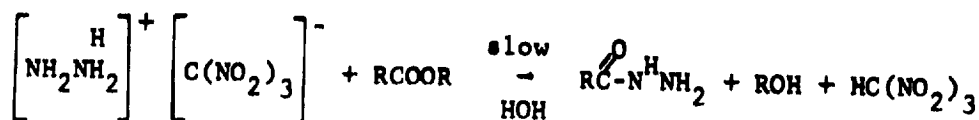
6. Metal Oxides



7. Carbonyls

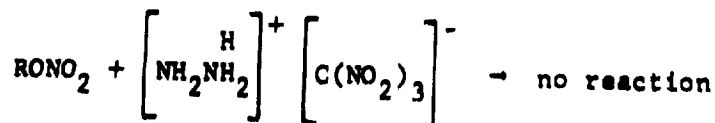


8. Esters



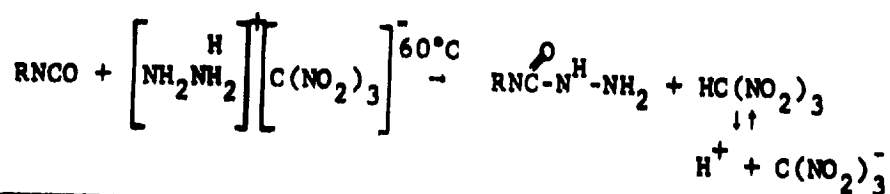
Rate of reaction depends upon ester.

9. NG + NC*



Stabilize each other.

10. Isocyanates



*U.S. Naval Propellant Plant. TMR-188. Hydrazine Nitroform, Its Synthesis and Properties. W. Blankenship, et al. June 1, 1961. Confidential

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dissolve the HNF. This third agent is commonly the glycol in most polyurethane binders. HNF and toluene diisocyanate have been mixed together and kept at 50°C for 144 hours without apparent reaction. However, when HNF and polypropylene glycol 2025 were mixed at 50°C, decomposition occurred within 10 hours.

As a result of the need to obtain high solid loadings and to modify the burning rate and pressure exponent of HNF-containing propellant systems, ultrasonic recrystallization was used to obtain more symmetrical (L/D=1) particles. Figure I-1 compares the as-received HNF and the recrystallized material under the same magnification. The length-to-diameter ratio was modified from approximately 10:1 to 1:1.

HNF PROPELLANT SYSTEMS

Table II presents a summary of the theoretical specific impulse at sea level and at altitude (expansion ratio at altitude of 50:1) and the theoretical flame temperature for all the major systems considered. Figure I-2 gives the theoretical vacuum specific impulse as a function of area ratio for different contents of JPL polyurethane binder.

Table III presents a general summary of the major HNF propellant systems on which work has been concentrated. As can be seen, only the TMETN double-base and TMETN-plasticized polyurethane systems have the processability, stability, and physical properties required. However, the ballistic properties of both systems were unacceptably high for the program requirements (r_b @ 700 psi = 0.23 in/sec, $n \leq 0.3$).

Shock Sensitivity

The aluminum analog (Arcocel 184) of the HNF-beryllium-TMETN plasticized double-base system was tested for shock sensitivity. A miniaturized card-gap test employing the standard apparatus normally used for card-gap testing of liquids was used. The test consists essentially of the detonation

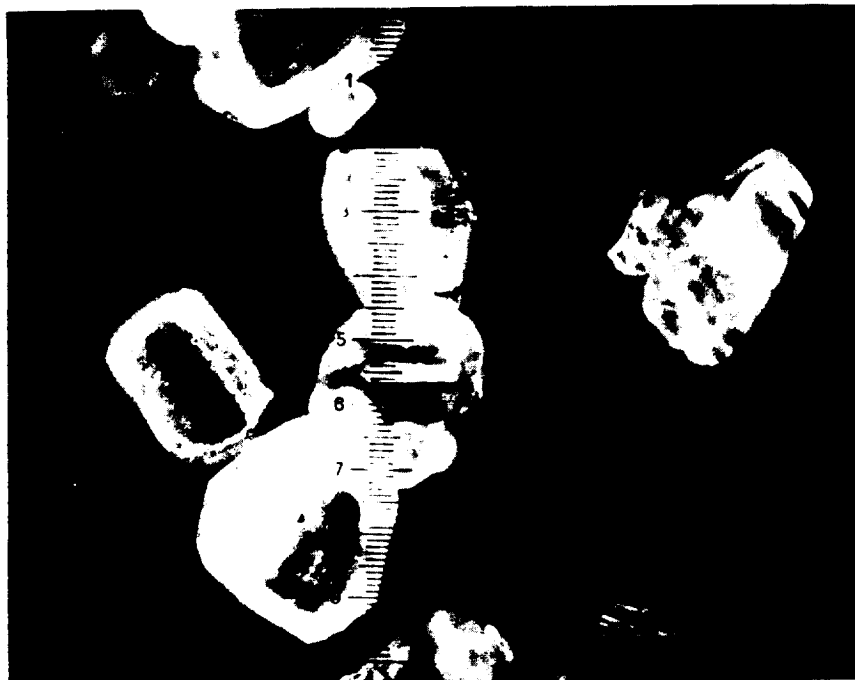
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TABLE II
SPECIFIC IMPULSE AND FLAME TEMPERATURE FOR
SEVERAL BERYLLIUM-CONTAINING SYSTEMS

<u>System</u>	<u>T_c</u> <u>(°K)</u>	<u>I_{1000/14.7}</u> <u>(lb-sec/lb)</u>	<u>I_{vac 50/1}</u> <u>(lb-sec/lb)</u>
25% PEG 200/NC HNF	3707	292.6	357
40% TMETN/NC HNF	4004	288.5	355
40% TMETN/DBS/NC HNF	3576	291.5	356
25% DBS/DBP/NC HNF	2527	291.2	350
27% DBS/DBP/NC HNF/AP	3327	285.9	344.5
35% TA/NC HNF	3519	285.1	348.5
40% TA/NC HNF	3214	280.0	337.5
18% PU(JPL) HNF	3410	293.3	-
20% PU(JPL) HNF	3275	291.5	-
22% PU(JPL) HNF	3140	288	-
22% PU/TMETN(1/1) HNF	3620	293.5	-
24% PU/TMETN(1/1) HNF	3580	293.3	-
26% PU/TMETN(1/1) HNF	3510	293.0	-

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Figure I-1. Comparison of HNF Particles as Received and Recrystallized.

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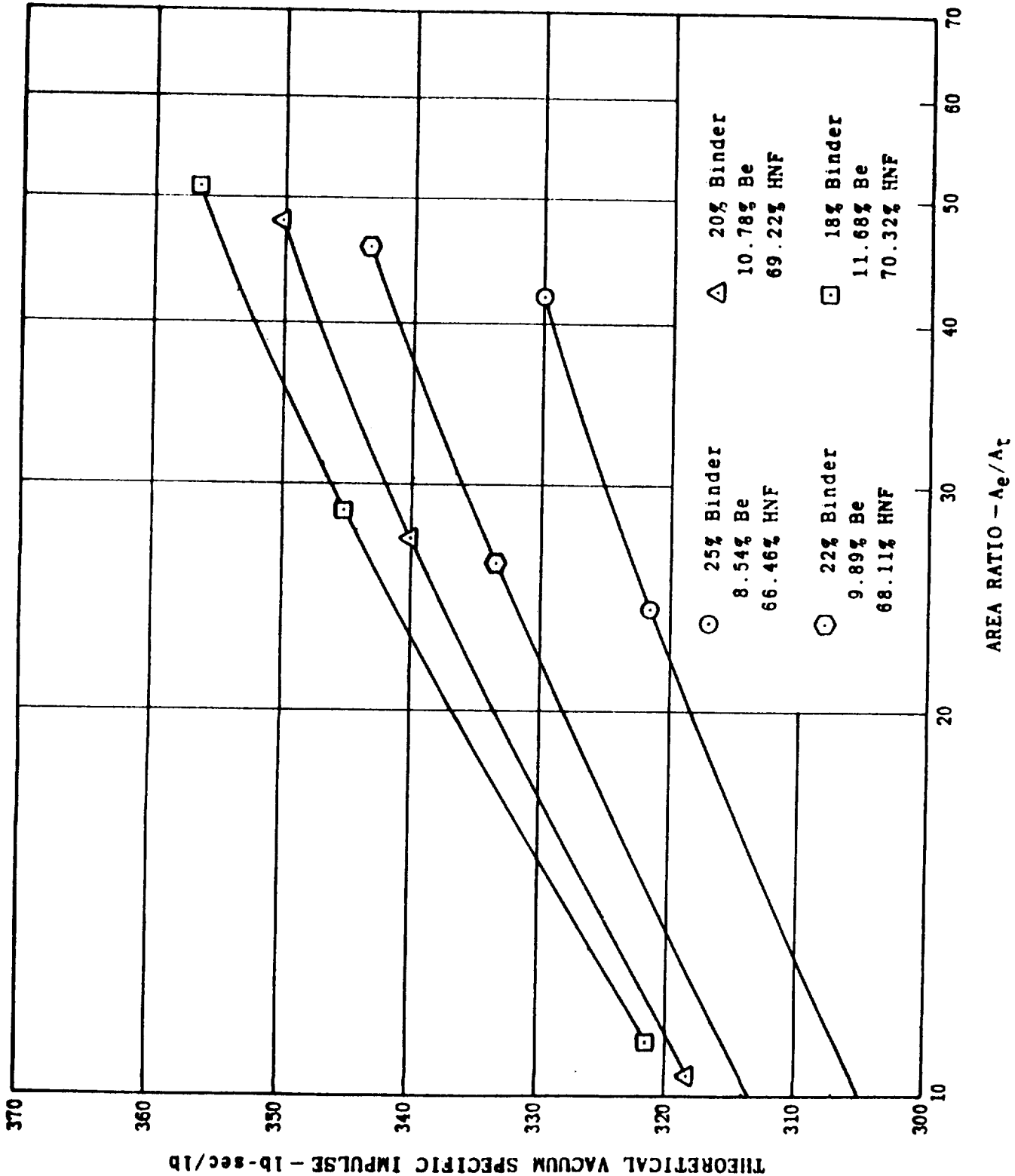


Figure I-2. Theoretical Vacuum Specific Impulse for Formulations of Polyurethane Binder (JPL), Beryllium, and Hydrazine Nitroform as a Function of Area Ratio.

TABLE III
PROPULSANT SYSTEM SUMMARY

PROPULSANT SYSTEM	STABILITY	PROCESSING	CURE	PHYSICALS	BALLISTICS	
					τ_b @ 700 psi	α @ 700 psi
A. TDSH/NC 40% Binder 32% Binder + 8% DMS	Good Good	Good Good	Good Good	Good Good	0.55 0.55	0.65 0.8
B. PDC/NC 25% Binder	Bad	Fair	Gases	-	0.5	1
C. DMS/DMP/NC 26% Binder (DMP/AP) 27% Binder (1% TDCM)	Fair Fair	Poor Poor	Poor Poor	Poor Poor	0.26 0.28	0.9 0.9
D. TA/NC 35% Binder 40% Binder 45% Binder	Poor Poor Poor	Poor Fair Good	Poor Poor Poor	Poor Poor Fair	0.28 0.26 0.20	0.5 0.5 0.3
E. POLYURETHANE 18% Binder 20% Binder 22% Binder	Good Good Good	Bad Poor Fair	- Good Good	- Poor Fair	- 0.35 0.32	- ≈ 1 ≈ 1
F. PU/TDSH (1/1) 22% Binder 24% Binder 26% Binder	Good Good Good	Fair Good Good	Fair Good Good	Poor Fair Good	0.6 0.55 0.55	≈ 1 ≈ 1 ≈ 1

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of a "booster" or "donor" explosive by a detonator and the attenuation of the resulting shock wave by a series of cellulose acetate cards placed between the donor and the acceptor. The number of cards placed between the donor and acceptor defines the "card gap" for that system. The actual determination of the detonation or nondetonation of the acceptor would require a measurement of the velocity of propagation of the shock wave through the acceptor charge. Since this is costly and difficult, the criterion for a detonation of the acceptor charge is established as the complete perforation of a steel witness plate placed above the acceptor charge. This criterion correlates adequately with actual velocity measurements. The card-gap test assembly is illustrated in Figure I-3. The detonator used in this case was a Corps of Engineers special blasting cap which was inserted into a cork base attached to the cardboard tube housing the assembly. On the cork base rested a tetryl pellet 1 inch high by 1-5/8 inches in diameter and weighing approximately 50 grams. The gap cards were cellulose acetate discs 1-5/8 inches in diameter and 0.01 inch thick. The container for the acceptor propellant charge was a section of nominal 1-inch schedule-40 steel pipe faced in a lathe to a length of 3.0 inches. Its inside surface was carefully cleaned by sandblasting to provide a good bond between the propellant and the wall of the container. Cork spacers were used to hold the sample tightly against the cards. Finally, the witness plate was a piece of cold-rolled, mild-steel plate 4 inches square by 3/8 inch thick.

The test proceeded generally as follows: the propellant was mixed, cast, deaerated, and cured by remote techniques in the processing bay. Each sample contained about 70 grams of propellant and was cured for about 2 hours at 55°C. Departing somewhat from standard explosives techniques because of the requirement for remote handling of the acceptor charge, the detonator was armed, tested for continuity, and placed in its cork holder in the cardboard housing with the cards and tetryl pellet inserted prior to being moved into the processing bay. The cardboard tube was then carefully placed on a portable steel pedestal mounted on an overhead trolley and remotely transported

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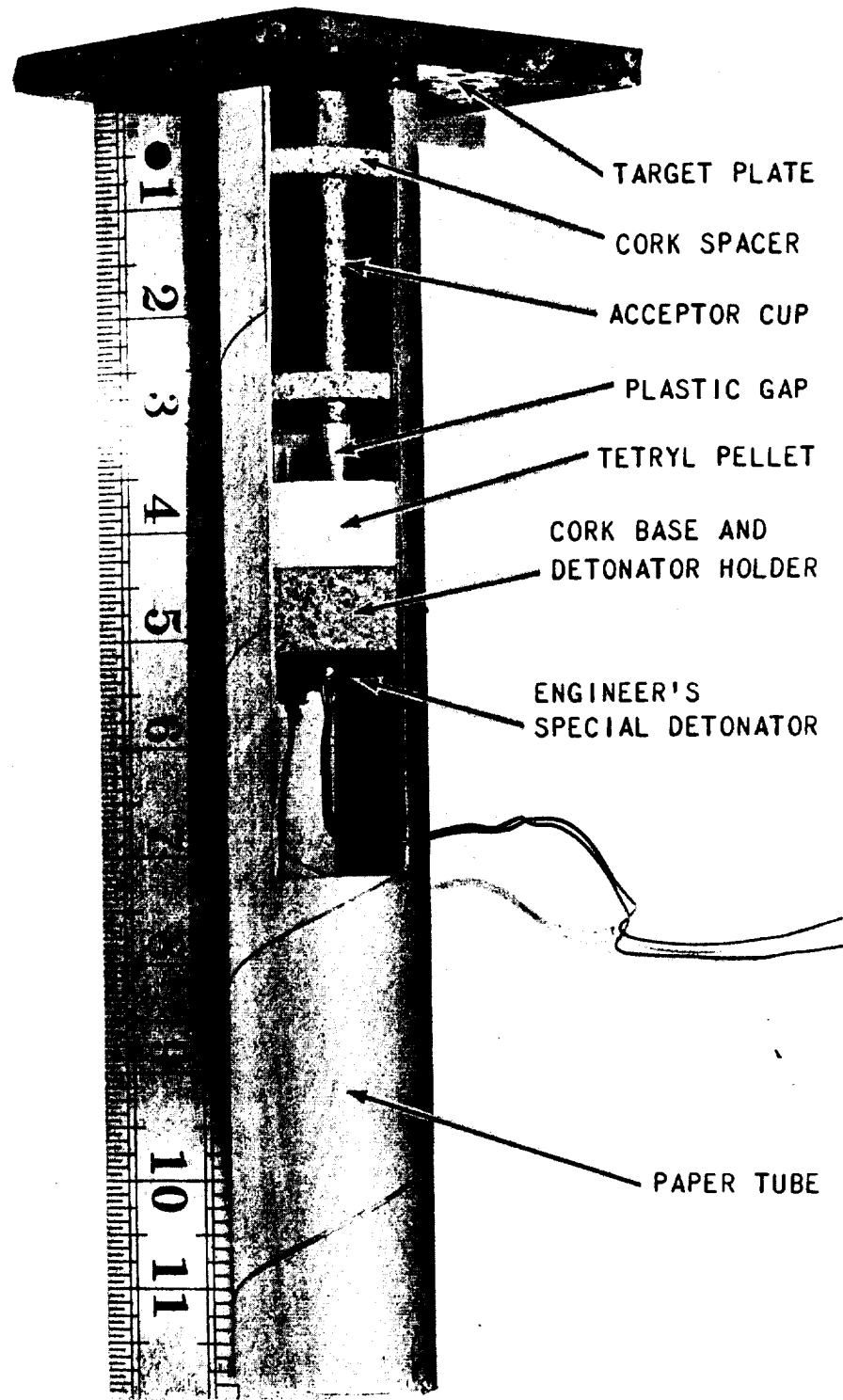


Figure I-3. Card-Gap Sensitivity Test Assembly.

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into the bay. Inside the bay, the acceptor charge and witness plate were placed on the assembly. The entire pedestal assembly was remotely transported to a $4 \times 4 \times 4$ foot pit lined with 1-inch steel plates and was remotely lowered into place. Finally, a trolley-mounted blasting mat woven from 5/8-inch-diameter cable was lowered over the pit to contain the shrapnel and deflect the shock wave of the detonation. Figure I-4 illustrates these procedures and pictures this equipment.

Twelve tests were performed with the Arcocel 184 formulation. Results are shown in Table IV. The data indicate a sensitivity for the Arcocel 184 formulation of about 195 cards, or 1.95 inches, at the 50-percent level.

Particle Size Effects

A study was conducted to determine the effect of the HNF particle size on the burning rate and pressure exponent of a propellant system. Table V summarizes the results and shows that the more symmetrical (L/D₅₀) and smaller the oxidizer particle, the lower the burning rate and pressure exponent. Substitution of beryllium seemed to increase both the pressure exponent and burning rate. With the recrystallized HNF, it has been possible to obtain similar burning rates and pressure exponents in double-base and some single-base formulations oxidized by either AP or HNF. This comparison is shown in Figure I-5 for a triacetin-plasticized nitrocellulose formulation using beryllium as the fuel.

Formulations and Test Results

The incorporation of HNF in a polyurethane binder necessitated the use of only prepolymer type binders. If a JPL X-535 type binder was used, the PPG-2025 dissolved some of the HNF which in turn reacted with the TDI. Even in the prepolymer type system, there was a tendency for the HNF crystals to absorb the prepolymer. This absorption resulted in a highly viscous mix.

With the use of a non-solvation agent such as TDI which preferentially wetted the HNF surface and resisted the absorption of the prepolymer,

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TABLE IV
RESULTS OF CARD-GAP TESTS ON ARCOCEL 184

<u>Test Number</u>	<u>Temperature</u>	<u>Card Gap</u>	<u>Result</u>
1	40°F	200	+ (positive)
2		300	- (negative)
3		250	-
4		225	-
5		200	-
6		190	+
7		195	-
8		190	+
9		195	+
10		200	-
11		195	+
12		200	-
Estimated 50-percent point		195	

Formulation of Arcocel 184

12.80 percent Aluminum
47.20 " HNF
13.30 " Nitrocellulose (12.6% N)
16.70 " TMETN
2.00 " TEGDN
4.00 " Dibutyl phthalate
4.00 " Dibutyl sebacate

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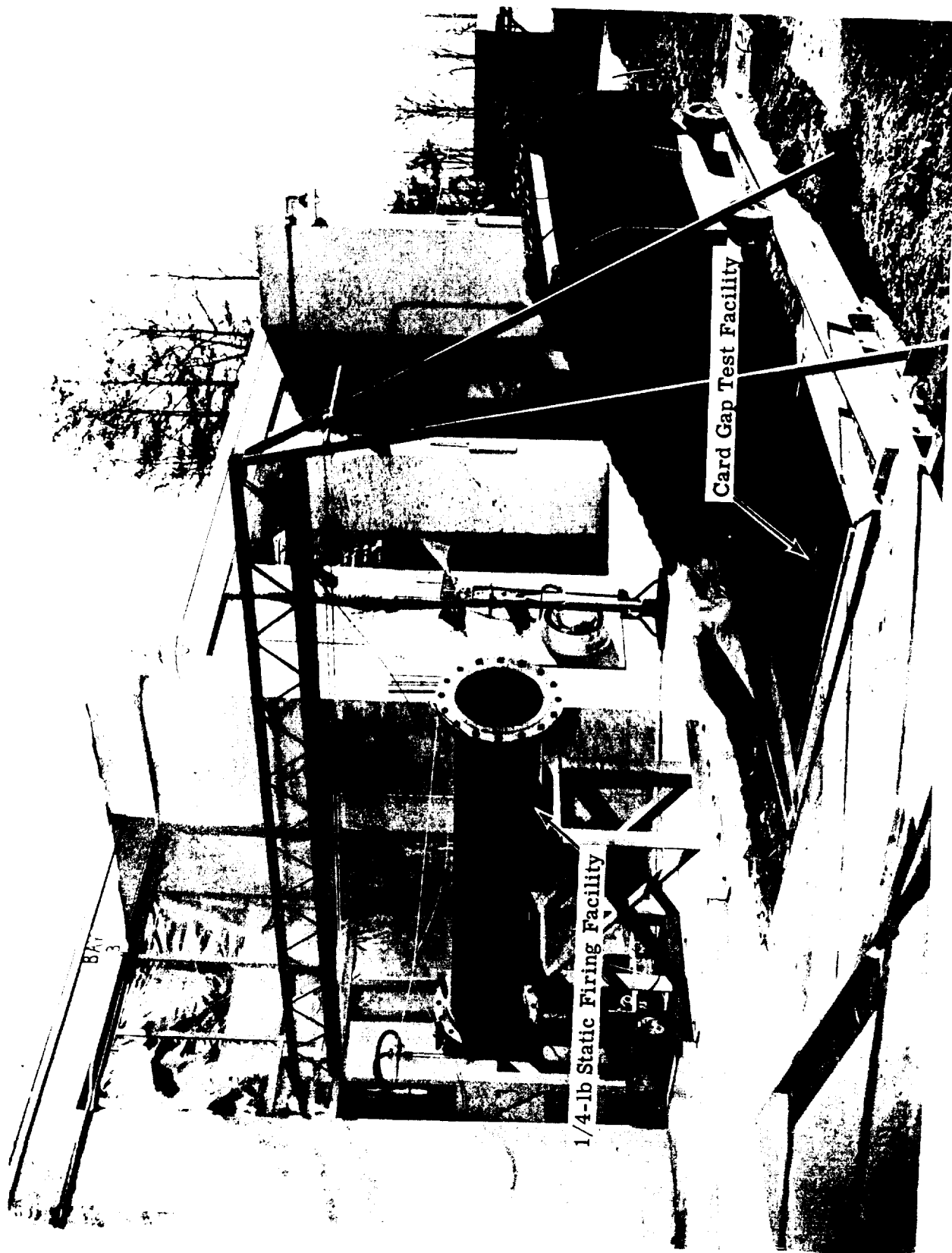


Figure I-4. Card-Gap Test Facility Behind Processing Building.

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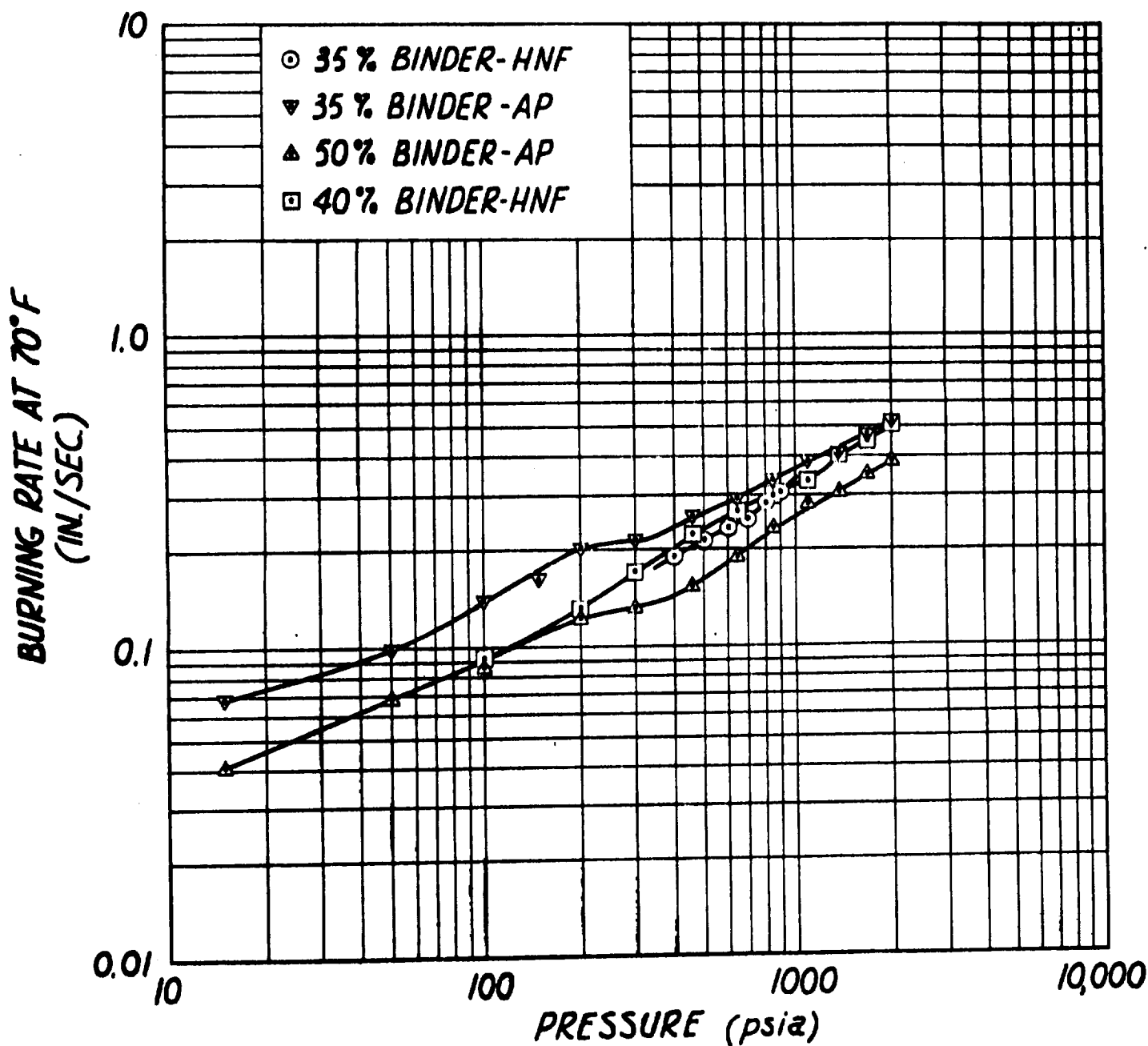


Figure I-5. Burning Rates for Formulations of Triacetin-Nitrocellulose Binder, Be, and Either HNF or AP.

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TABLE V
EFFECT OF PARTICLE SIZE ON BURNING
RATE AND PRESSURE EXPONENT

<u>Hydrazine Nitroform Particles</u>	<u>Burning Rate @700 psi (in/sec)</u>	<u>Pressure Exponent @700 psi</u>
200 μ (L/D \approx 10)	0.36	1.0
50 μ (L/D \approx 2)	0.23	0.76
200 μ /50 = 1:1 Mix	0.28	0.89

<u>Formulation</u>	<u>Percent</u>
Nitrocellulose	10.0
Dibutyl Sebacate	12.0
Dibutyl Phthalate	5.0
Aluminum ^a	16.9
Hydrazine Nitroform	56.1

^a Substitution of beryllium for aluminum increases both burning rate and pressure exponent.

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a castable aluminum-containing mix at the 20 percent binder level was made. However, the incorporation of beryllium at this binder content level resulted in an uncastable system.

A new approach was taken by the use of a non-glycol prepolymer. The prepolymer was made from castor oil and TDI with castor oil also being used as the cross-linking agent. FeAA was effective as a catalyst in this system. At 20 percent binder content, a beryllium-fueled mix was still too viscous to be castable by standard techniques.

It was found that trimethylolethane trinitrate (TNETN) was compatible with the binder components being used. Two 1/4-pound motors¹ were cast with an aluminum-fueled propellant containing 26 percent of nitro-plasticized polyurethane binder. The binder-to-plasticizer (TNETN) ratios were 3:1 and 1:1. These motors were cured at 50°C for 18 hours. When fired, both motors ignited, reached a 550 psi chamber pressure, lost pressure, and burned out at ambient pressure. The strand ballistic data showed a pressure exponent of 1 for both motor formulations. With the particular geometric configuration and formulation ballistic properties, the effect of a high pressure exponent would be for the motors to ignite and then reduce pressure to ambient rather than to explode. In the equation:

$$P_c = \left(\frac{KA_B \rho}{A_t C_D} \right)^{\frac{1}{1-n}}$$

where:

P_c = chamber pressure

K = burning rate constant

A_B = burning surface area

A_t = throat area

¹Formulation shown on Figure I-5.

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C_D = discharge coefficient

n = pressure exponent

ρ = propellant density

the term $\left(\frac{K A_B \rho}{A_t C_D} \right)$ was slightly less than one for both motors. With $n \geq 1$ and $\left(\frac{K A_B \rho}{A_t C_D} \right) < 1$, the chamber pressure approaches zero.

A series of additives was investigated in an effort to reduce the pressure exponent to an acceptable level. Some of the additives tried were ammonium oxalate, lithium fluoride, and a cab-o-sil coating of the oxidizer particles. Both ammonium oxalate and lithium fluoride reduced the pressure exponent from one to between 0.6 and 0.7 at the 2-percent level. However, the theoretical sea-level specific impulse was also reduced to around 285 lb-sec/lb. The replacement of 50 percent of the HNF by ammonium perchlorate only lowered the theoretical specific impulse to 287 lb-sec/lb and reduced the pressure exponent to 0.55 at 500 psi.

Two aluminized 1/4-pound motors were again cast with a binder-to-plasticizer ratio of 1:1 and 26 percent of total plasticizer and binder. Both motors fired successfully. Only the chamber pressure was recorded for both firings.

Both motors fired at a chamber pressure approximately half that of the designed pressure. The chamber pressures were 185 psi and 375 psi. A comparison of the motor and strand burning rates is shown in Figure I-6. The motor burning rates are higher than the strand burning rates at the same pressure, but the pressure exponent appears to be somewhat lower in the motors. However, more motor data are needed before this trend can be confirmed. The theoretical discharge coefficient for the formulation was 0.00622. The measured discharge coefficients were 0.00686 and 0.00704. The C_D efficiencies were 90.7 percent and 88.4 percent. The pressure trace of one of the firings is shown in Figure I-7. There was a 0.16-second delay between the ignition pressure peak and the development of full chamber pressure. No residue was found in the motor after firing.

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CONCLUSIONS

The work on HNF systems performed under this program has led to the following general conclusions about the nature of hydrazine nitroform propellants:

(1) TMETN-plasticized double-base formulations with HNF oxidizer appear to have the best over-all stability, processability, physical properties and ballistic properties.

(2) HNF can be incorporated in a polyurethane binder to give a thermally stable system provided that the polyurethane is a non-glycol (such as castor oil) TDI-prepolymer-type binder.

(3) In polyurethane and nitroplasticized-polyurethane formulations where there is a high HNF solids loading, the pressure exponent approaches 1 in the strand ballistic data.

(4) The presence of water hydrolyzes almost any ester-type nitrocellulose plasticizer, which results in the degradation of HNF in the formulation.

(5) The presence of ferric oxide and moisture as an impurity in HNF will result in a catalytic oxidation-reduction degradation of the HNF in which both NO and N_2O are given off.

(6) The as-received needle-shaped ($L/D \approx 10$) HNF can be ultrasonically recrystallized to obtain more symmetrical particles ($L/D \approx 1$). This allows much higher solids loadings and better formulation processability.

(7) The more symmetrical ($L/D \approx 1$) and the smaller the HNF oxidizer particle, the lower the burning rate and the pressure exponent.

(8) Substitution of beryllium for aluminum in an HNF-oxidized system appears to increase both burning rate and pressure exponent.

(9) Motor burning rates of HNF-oxidized, nitroplasticized polyurethane propellants appear to be higher than the strand burning rates.

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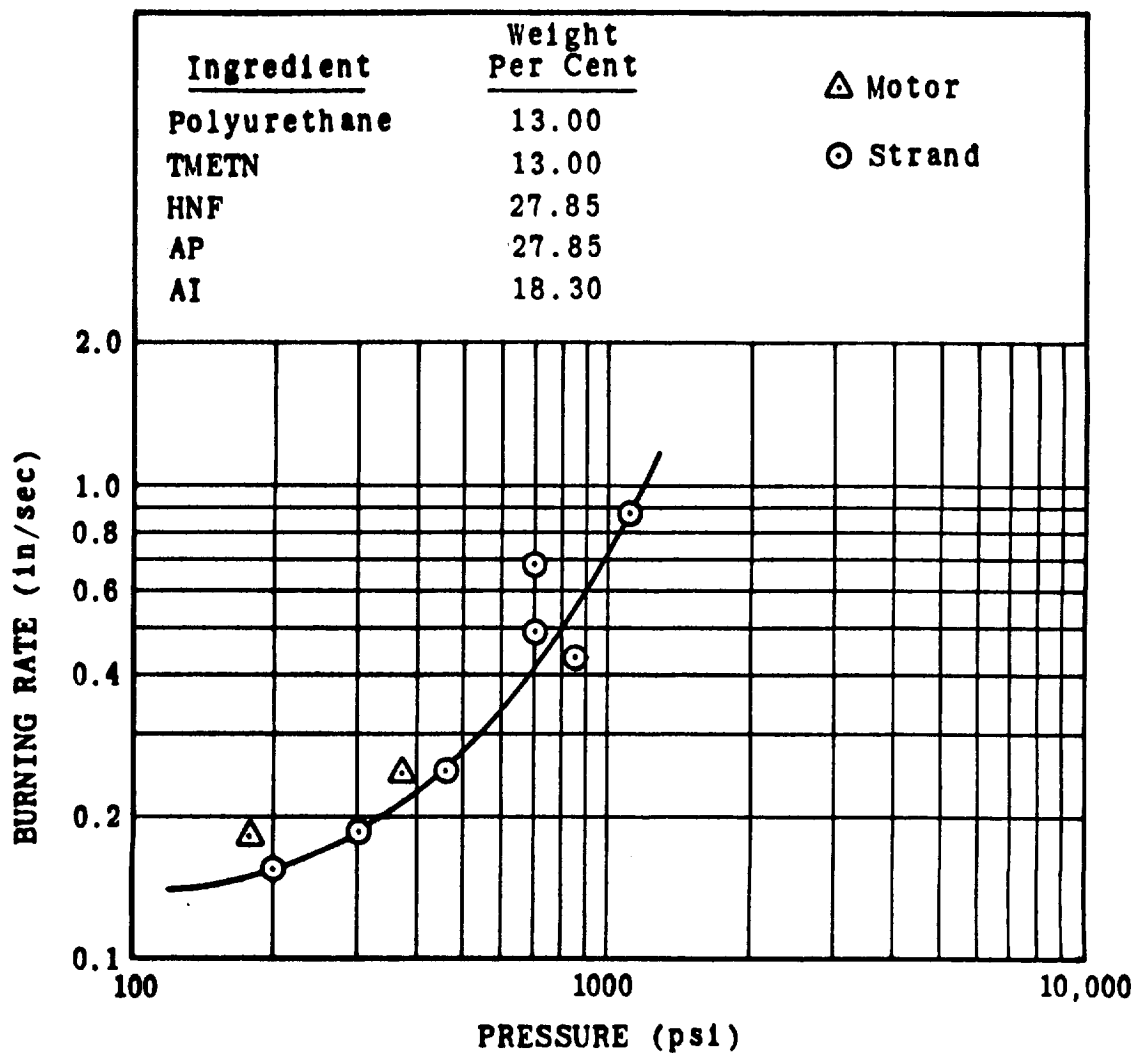


Figure I-6. Motor and Strand Ballistic Data.

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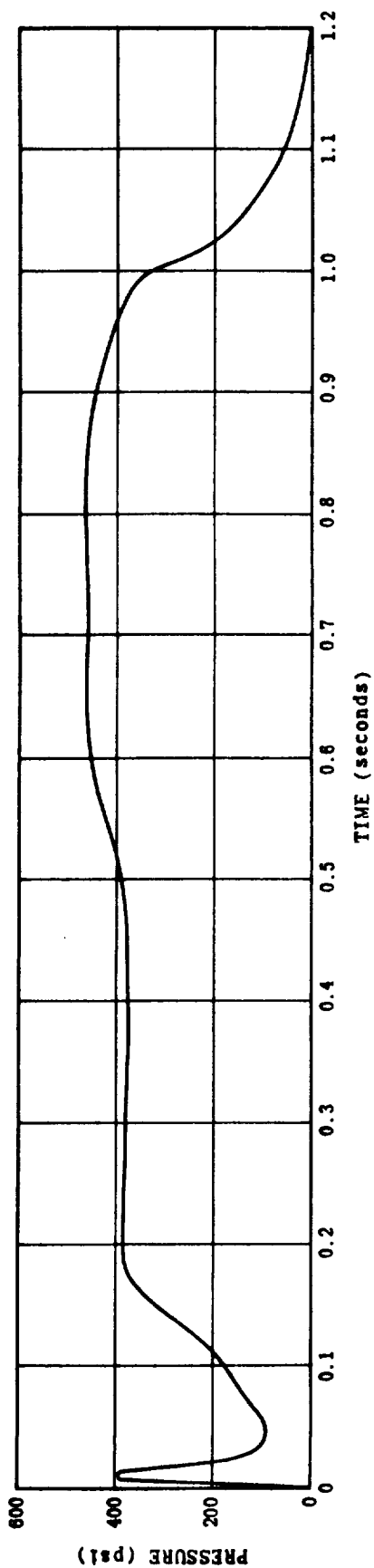


Figure I-7. One-Quarter Pound Motor Pressure Trace.

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(10) Lithium fluoride and ammonium oxalate appear to be effective pressure-exponent depressants at the 2-percent level for HNF-polyurethane systems, but degrade the thermodynamic performance to an unacceptable level.

(11) Substitution of ammonium perchlorate for HNF appears to reduce the pressure exponent, burning rate, and thermodynamic performance proportional to the amount of HNF replaced.

(12) The theoretical thermodynamic performance, stability, processability, physical properties and ballistic properties of a TMETN-plasticized polyurethane (1:1 ratio) propellant oxidized with 50 percent HNF and 50 percent ammonium perchlorate is equivalent to a TMETN-plasticized double-base formulation oxidized by HNF alone.

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II. TAILORING AND TESTING OF POLYURETHANE PROPELLANT

BACKGROUND AND INTRODUCTION

Atlantic Research Corporation has participated in polyurethane propellant research and development for several years. The original beryllium-containing propellant, developed under Air Force Contract No. AF 33(161)-6623, was a prepolymer-based polyurethane formulation. In the preliminary work, with beryllium-containing formulation safety was of primary importance, but as familiarity with techniques for handling beryllium powders was gained, it became possible to use more complex binders. Since the physical properties of the prepolymer-based propellants were not optimum, improved binders were required.

Early in 1961, Atlantic Research began to use the JPL X535 polyurethane binder. These propellants are easily processed and have adequate pot life; the cured materials have excellent physical properties. They have been static fired in 10- and 50-pound cylindrical and 50-pound spherical motors. This work was conducted under an Air Force contract (AF 04(611)-7017) with the objective of tailoring beryllium-containing polyurethane propellants for upper-stage applications. The program began in March 1961 and continued through the end of that year.

This program was conducted concurrently with a program (Contract AF 04(611)-7037) for delivery of six 17-inch spherical motors of beryllium-containing polyurethane propellant—three for static firing and three for use as fourth stage of the Blue Scout Junior vehicle. To meet static testing and delivery schedule requirements, these motors were loaded with a prepolymer-based beryllium-containing polyurethane propellant which had been developed by Atlantic Research. Subsequent to the selection of the propellant for the Blue Scout Junior application, the tailoring program resulted in an improvement in the binder physical properties of the polyurethane system. Investigations of the effect of binder content, beryllium particle size, and thrust level on specific-impulse efficiency also continued.

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Binder physical properties were improved by the use of JPL-type polyurethane binders. Tensile strengths of 100 psi with elongations of 100 percent were measured at room temperature with beryllium propellants containing 20 percent of the JPL binder.

The results of static firings in this program are summarized in Table I. The data obtained from the 50-pound cylindrical motor firings are not believed to be valid. The inconsistencies in the data are believed to have been dynamic effects on the load cell due to the rapid and extreme pressures generated in the closed firing tunnel by a motor of this size.

A summary of physical property data from this program is presented in Table II. Typical values for propellants with 20 or 22 percent prepolymer binders are 60 to 70 psi tensile strength, 30 to 40 percent ultimate elongation, and 300 to 600 psi Young's modulus.

In the discussion of HNF propellants it was pointed out that in late 1961 it was decided to shift development effort for the 17-inch spherical motor to a polyurethane propellant. A discussion of the tailoring and testing of this propellant follows; the work described was carried out at the Atlantic Research facilities described in Appendix B of this report.

POLYURETHANE PROPELLANT DEVELOPMENT

Propellant Tailoring Program

The tailoring program proposed included both 10- and 50-pound motor firings during the final quarter of 1962. There were four distinct phases:

- (1) aluminum-containing control rounds
- (2) beryllium powder evaluation
- (3) confirmation firings
- (4) firings to determine the effect of scale-up

Phase 1 involved the fabrication and static firing of ten aluminum-containing polyurethane control rounds which would be made throughout the 10-pound program. These grains would provide controls for comparison with the

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TABLE I
SUMMARY OF STATIC FIRING RESULTS
OBTAINED ON CONTRACT AF 04(611)-7017

10-POUND FIRINGS										50-POUND FIRINGS									
ARCANE 30 ^a (20% aluminum)					ARCANE 38 (12.5% beryllium)					ARCANE 39 (13.5% beryllium)					ARCANE 38 (12.5% beryllium)				
Oxidation Ratio ^b : 1.11					Oxidation Ratio ^b : 1.09					Oxidation Ratio ^b : 1.09					Oxidation Ratio ^b : 1.09				
Firing Number	217	223	225	218	228	224	229	230	220 ^c	221 ^c	219 ^d	222 ^d	221 ^c	219 ^d	222 ^d	221 ^c	219 ^d	222 ^d	233 ^d
Date Fired	10/3/61	10/16/61	10/17/61	10/4/61	10/30/61	10/16/61	10/31/61	10/31/61	10/31/61	10/16/61	10/31/61	10/31/61	10/16/61	10/31/61	10/31/61	10/16/61	10/31/61	10/31/61	10/31/61
Propellant weight, lb	11.51	11.52	11.52	10.625	10.90	11.05	10.85	11.04	45.24	45.32	48.74	49.67	48.91	48.91	48.91	48.91	48.91	48.91	48.91
Expansion ratio, A/A _t	12.10	11.96	9.49	5.91	5.98	5.62	5.61	5.44	3.86	3.80	7.92	7.90	7.80	7.80	7.80	7.80	7.80	7.80	7.80
Action time, 10%-10%, sec	4.422	4.530	4.528	3.887	3.711	3.217	3.274	3.311	12.92	13.44	4.95	4.97	5.15	5.15	5.15	5.15	5.15	5.15	5.15
Average chamber pressure (action time), psia	755	734	918	678	741	859	786	763	653	613	1097	1102	1037	1037	1037	1037	1037	1037	1037
Average ambient pressure (action time), psia	13.89	13.61	14.43	14.43	14.15	14.18	15.24	13.93	13.73	12.24	17.32	14.51	27.70	27.70	27.70	27.70	27.70	27.70	27.70
Average burning rate (action time), in/sec	0.235	0.229	0.230	0.267	0.279	0.324	0.318	0.330	0.302	0.256	0.383	0.302	0.291	0.291	0.291	0.291	0.291	0.291	0.291
Average thrust (action time), lb	580	570	590	725	720	840	810	815	830	790	2700	2925	2350	2350	2350	2350	2350	2350	2350
Measured I _{sp} ^e , lb-sec/lb																			
Analog 1	224.4	224.4	232.4	233.5	245.6	245.6	244.6	244.8	237.3	235.1	274.7	293.8	251.5	251.5	251.5	251.5	251.5	251.5	251.5
Analog 2	224.1	224.3	232.0	233.1	243.2	244.5	243.5	244.0	237.4	235.6	273.9	292.5	249.0	249.0	249.0	249.0	249.0	249.0	249.0
Digital 1	225.0	226.3	233.2	232.4	244.8	246.3	245.4	245.8	235.3	232.5	280.5	297.4	252.4	252.4	252.4	252.4	252.4	252.4	252.4
Digital 2	224.7	226.1	232.5	236.7	243.8	245.3	244.5	244.4	234.5	233.0	280.4	295.5	250.7	250.7	250.7	250.7	250.7	250.7	250.7
Average	224.5	225.3	232.5	233.9	244.4	245.4	244.5	244.8	236.1	234.0	277.4	294.8	250.9	250.9	250.9	250.9	250.9	250.9	250.9
Theoretical I _{sp} ^f , lb-sec/lb	253.1	252.9	259.0	268.3	271.0	272.2	269.5	270.1	262.3	262.7	277.6	281.1	263.2	263.2	263.2	263.2	263.2	263.2	263.2
Average I _{sp} efficiency, %	88.7	89.1	89.0	87.3	90.2	90.1	90.7	90.6	90.0	89.1	99.9	104.8	95.3	95.3	95.3	95.3	95.3	95.3	95.3

- a. Formulations of Arcane propellants may be found in Appendix B.
b. Oxidation ratio is defined as moles of oxygen divided by the moles of carbon plus the moles of beryllium. A formulation stoichiometric to CO and BeO has an oxidation ratio of 1.00.
c. 12-inch-diameter spherical motor.
d. 10-inch-diameter cylindrical motor 10C7-21.
e. Fifteen degree cone angle.
f. Computed at the firing conditions, 0 degree cone angle.
g. Measured I_{sp} times 100 divided by the theoretical I_{sp} at firing conditions.

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TABLE II

PHYSICAL PROPERTIES OF POLYURETHANE PROPELLANTS
CONTAINING ALUMINUM OR BERYLLIUM AND OXIDIZED WITH AMMONIUM PERCHLORATE.

<u>Arcane Number</u>	<u>Metal</u>	<u>Binder Type</u>	<u>Binder Content (percent)</u>	<u>Physical Properties at 70°F</u>		
				<u>Ultimate Tensile (psi)</u>	<u>Ultimate Elongation (percent)</u>	<u>Young's Modulus (psi)</u>
30	Al	JPL	20	130	70	200
38	Be	JPL	20	140	50	500
39	Be	JPL	18	100	60	600

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various beryllium-containing polyurethane propellants being evaluated. The propellant chosen was Arcane 42, an OMOx composition containing 18 percent of JPL polyurethane binder. This propellant is easily processed and has good physical properties. A total of ten grains was scheduled, five to be fired in the Atlantic Research Corporation controlled-atmosphere tunnel and five to be fired under normal sea-level conditions. All firings were to be conducted at a chamber pressure of approximately 1,000 psi with an optimum nozzle expansion ratio.

Phase 2 was intended to select the beryllium powder to deliver the highest performance possible. The propellant chosen was Arcane 40, the OMOx beryllium-containing composition with 18 percent of JPL polyurethane binder. Twenty-six firings were scheduled in the Atlantic Research tunnel, all at approximately 1,000-psi chamber pressure with optimum expansion ratio. These firings were divided as follows:

(1) Three firings with "17-micron" beryllium powder (Brush Beryllium Corporation). This material has been used in other programs at Atlantic Research and considerable background information about properties and particle size exists.

(2) Three firings with "400-mesh" beryllium powder (General Astro-metals Corporation). This beryllium powder is ground from chips or flakes made in France by the Pechiney process. It has not been evaluated extensively, but the analytical data available indicate that it is a high-purity product worthy of evaluation.

(3) Three firings with Brush Beryllium Corporation's "17 \pm 5 micron" or 1755 beryllium powder. This material is classified from the standard "17-micron" powder and is a closer cut. All fine and large powder are removed. The lack of fine powder is an aid to processing and the removal of large particles improves combustion efficiency.

(4) Nine firings with Berylco low-oxide-content beryllium powder (Beryllium Corporation of America). This material is similar to Brush "17-micron" beryllium powder. Three grades are available, which have 0.6, 1.0,

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or 2.0 percent of oxide. Three firings of each oxide content would be required to assess the effects of oxide content on specific-impulse efficiency. This would also ensure that there was no appreciable difference in the viscosity characteristics of propellants made from these powders.

(5) Eight firings with varying percentages of "325-mesh" beryllium powder (Brush Beryllium Corporation). This powder is somewhat larger in particle size than the Brush "17-micron" but some optimum blend may exist. Two firings each were scheduled of propellants containing "325-mesh" and "17-micron" in ratios of 100:0, 75:25, 50:50, and 25:75. The three firings of all "17-micron" under (1) above would serve as 0:100 controls for these eight firings.

At this time it was thought that a selection could be made and Phase 3 would be undertaken. Phase 3 would consist of five to ten sea-level firings and at least ten firings at simulated altitude conditions to demonstrate the delivered specific impulse of the selected formulation. Most of the sea-level firings would be at a chamber pressure of 700 psi with optimum expansion ratios. Firings under simulated altitude conditions would be at expansion ratios of 50:1 and 60:1.

Phase 4, the evaluation of scale-up effects, was scheduled to be carried out concurrently with Phase 3. Three firings were recommended in a double-length motor to evaluate the effect of a change in mass-flow rate and thrust level at a chamber pressure of 700 psi. Also two 50-pound motors were scheduled. Aluminum-containing control grains of the same configurations were scheduled for static firing both in the Atlantic Research tunnel and on an outdoor thrust stand.

When this program was outlined, it became apparent that not all of the objectives could be met and still produce a qualified propellant in time to phase-in with the 17-inch spherical motor program. As a result of this, and after discussion with Jet Propulsion Laboratory personnel in late January, the program was modified to consist of:

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- (1) Two static firings of Arcane 42, an aluminum-containing propellant.
- (2) Ten static firings of Arcane 40 modifications, a beryllium-containing propellant, for evaluation purposes.
- (3) Fifteen firings of the final propellant for demonstration of delivered specific impulse.
- (4) Seven additional firings for $P-K_n$ data.

The ten static firings of Arcane 40 types were to include two firings each of four variations of Arcane 40 and two firings of Arcane 39, an oxidizer-rich, beryllium-containing propellant with 18 percent of binder. The four varieties of beryllium powder to be used in Arcane 40 were Brush "17-micron", General Astrometals "400-mesh", Berylco "17-micron (0.6 percent BeO)", and Brush "D₅₀ equal to or less than 10 microns". All of these except the latter have been discussed previously. This is a special cut which is classified from normal Brush "17-micron" until the weight average particle diameter is less than 10 microns at the 50-percent point.

The best of these four variations was to be used in making two Arcane 39 grains. Evaluation of the results of the static firing of these two grains would permit a selection of Arcane 39 or Arcane 40, on the basis of delivered specific impulse, specific-impulse efficiency, and burning rate.

The selected propellant would then be qualified by five firings at sea level with an optimum expansion ratio, and ten firings at altitude with a 50:1 expansion ratio. Five propellant batches of three grains each would permit the firing of one grain at sea level and two at altitude from each batch.

At this point, or perhaps during the qualification, the propellant would be ready for casting of a 17-inch spherical grain. Further work would be limited to firing seven grains of a 6C2-11.4 configuration to obtain $P-K_n$ data. These would be accomplished as follows:

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<u>Temperature, °F</u>	<u>Pressure Range, psia</u>		
	<u>300-900</u>	<u>400-1200</u>	<u>500-1500</u>
130	1	1	
70	1	1	1
20	1	1	

The described program anticipated qualification in February 1962. At this time, difficulties arose in processing the beryllium-containing polyurethane propellant. This will be discussed further in a subsequent section; however, they posed a great problem from a program schedule standpoint. Because of the long delay caused by propellant processing problems, it was decided to cast the first 17-inch motor before the propellant tailoring program was completed. The program was completed, as outlined, with the exception of the P-K_n firings. At the conclusion of the simulated altitude firings, it was decided to omit the P-K_n firings and concentrate the effort on the 17-inch motor fabrication.

Propellant Formulation and Processing

Binder

The original polyurethane work on this program utilized JPL formulation containing PPG 2025-one Alrospense 11P:Trimethylolpropane in an equivalents ratio of approximately 0.81:0.10:0.09. The isocyanate-to-hydroxyl ratio (NCO:OH) was higher than the 1.05 NCO:OH ratio considered optimum by JPL. In calculating NCO:OH ratios, the active hydrogens on phenyl-β-naphthylamine (Neozone D), were included, whereas JPL neglected these, feeling they were so hindered as to be virtually unreactive. Also, at Atlantic Research, component analyses were used solely for formulating. As a consequence of these differences, Atlantic Research cures were somewhat unreliable. Aluminum-containing formulations showed good curing properties; two spherical motors, 17S-1 and 17S-2, were cast and cured successfully in late 1961 and early 1962. Beryllium-containing polyurethane propellants generally did not cure with two exceptions: a mix made with Brush "325-mesh" beryllium in January, 1962, which was too viscous to process successfully, did cure satisfactorily; another, made with Brush "17 ± 5 micron" beryllium, is discussed below.

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Discussions with JPL in February pointed out the differences in our calculations. Formulating to an NCO:PPG:Alrospense 11P:TMP equivalents ratio of 1.05:0.78:0.11:0.11 and ignoring the hydroxyl content of phenyl- β -naphthylamine gave polyurethane binders and aluminum-containing propellants which cured reliably. The number of equivalents of each ingredient was calculated using equivalent weights of 87.1 for toluene diisocyanate, 45.2 for trimethylolpropane, and 208 for Alrospense 11P. The equivalent weight for polypropylene glycol 2025-one was calculated from the hydroxyl number supplied by the manufacturer, assuming an average molecular weight of 1,950. Cures of beryllium-containing propellants were still unreliable; however, some Brush "17 \pm 5 micron" beryllium (lot 23R) was used in three successive mixes of Arcane 40Y, two mixes with our ingredients and formulations (equivalents ratio, 1.04:0.811:0.096:0.093) and one mix with ingredients and formulation supplied by JPL (equivalents ratio, 1.05:0.78:0.11:0.11). All cured better than anything that had been made previously, and the 10-pound grain made from JPL materials was adequate for static firing. These three mixes exhausted the supply of lot 23R. Soon afterwards, the batch of normal "17-micron" beryllium we were currently employing, 23P, was returned to the manufacturer for exchange and 100 pounds of a new lot, batch 11, were obtained.

Propellant formulated from batch 11 of normal 17-micron beryllium gave reliable cures in the motors without exception; however, sheets of propellant made in the laboratory or the propellant processing area did not cure reproducibly. This is not surprising if one considers the relative surface-to-volume ratios in the two cases—15:1 for the sheet as compared with about 2:1 for a spherical propellant charge. The physical property and burning rate sheets were cured in flat molds on which the propellant was spread into a layer of the appropriate thickness (0.075 inch for physical properties test sheets). Attempts were made to cure the sheets in a number of ways, e.g., covered and uncovered, at temperatures from 50 to 80°C, and in ovens or Carver laboratory presses. None of these methods was effective.

So much trouble had been encountered prior to receipt of batch 11 that when subsequent lots of beryllium were received, it was decided to evaluate them immediately on receipt to determine their usefulness. On the basis

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of the above problems encountered in obtaining reliable cures in sheets of propellant, it was decided to evaluate each lot of beryllium in the propellant processing area instead of the laboratory. This was done in 2,000-gram mixes, to minimize the time, labor, and materials involved. These 2,000-gram mixes were cast into containers shaped from aluminum foil, approximately 4.25 inches in diameter and 5 inches high. These have a surface area of about 100 square inches and a volume of about 75 cubic inches of propellant, thus approximating the surface-to-volume ratio of 10-pound grains.

Using these mixes, it was determined that none of three subsequent 100-pound lots of Brush "17-micron" beryllium, batches 25, 34, or 45, cured as well as batch 11. This evaluation was based on hardness and degree of thermoplasticity after an overnight (approximately 16 hours) cure in an oven at 60°C. Although this is only a qualitative evaluation, it has been substantiated by the appearance of grains cast from propellants made with these lots of beryllium. None of these grains cured as well as grains made from propellant containing batch 11 beryllium.

Throughout these experiments, surveillance of binder ingredients was maintained. In addition, each binder batch made was sampled and fully cured, acceptable samples were obtained of inert binder and of aluminized propellant (Arcane 42) containing the binder before it was used to make beryllium-containing propellant.

Oxidizer

The background work described in Section I had shown that the propellant burning rates of beryllium-containing polyurethane propellants formulated with JPL X535-type polyurethanes were higher than those of analogous propellants based on a plasticized prepolymer-type polyurethane binder¹. A lower burning rate was required to meet the motor design requirements.

¹Atlantic Research Corporation, Tailoring of High-Energy Propellants, Final Report. Contract AF 04(611)-7017, November 1961. CONFIDENTIAL

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The first laboratory work investigated the lowering of burning rates by two methods: (1) incorporation of RDX (cyclohexamethylenetrinitramine) as part of the oxidizer and (2) use of ammonium perchlorate with extra-large particle size. The following results were obtained.

<u>Arcane</u>	<u>Oxidizer^a</u>	<u>Oxi- dizer Ratio</u>	<u>Binder Content (percent)</u>	<u>Metal Content (percent)</u>	<u>r₆₀₀ (in/sec)</u>	<u>n₆₀₀</u>
38	RDX/Unground NH ₄ ClO ₄	1:9	20	13.05	0.225	0.33
38	RDX/Unground NH ₄ ClO ₄	1:6	20	12.31	0.22	0.23
38	RDX/Unground NH ₄ ClO ₄	1:3	20	11.57	0.215	0.20
40 Mod	A/B	5:2	18	14.95	0.255	0.012
40Y	C/B	5:2	18	14.95	0.212	0.25
40	D	7:3	18	14.95	0.255	0.25

- ^a
- A = Ammonium perchlorate, American Potash & Chemical, -20+35 mesh, spherical.
 - B = Ammonium perchlorate, Pacific Engineering Corp., Class I spherical.
 - C = Ammonium perchlorate, Pacific Engineering Corp., -10+48 mesh, spherical.
 - D = Ammonium perchlorate, unground, 24-mesh and 2TH 6900 rpm.

Arcane 40, an OMOx composition with less binder and a higher flame temperature than either Arcane 38 or Arcane 39, was thought to be the composition capable of delivering the highest specific impulse. The original propellant work showed that Arcane 40, containing a 7:3 blend of unground and 2TH 6900 rpm grind ammonium perchlorate, would have a motor burning rate that was slightly higher than desired. Further experiments showed that a 5:2 blend of Pacific Engineering Corporation's -10+48 mesh and class I spherical ammonium perchlorates (Arcane 40Y) lowered the burning rate significantly. Arcane 40Y contains the maximum amount of -10+48 mesh ammonium perchlorate possible, commensurate with reasonable processability. However, propellants made with this oxidizer mixture were somewhat difficult to process. RDX-ammonium perchlorate mixtures were also satisfactory, but the use of large ammonium perchlorate was considered to be the best choice. A 2:2:1 blend of -10+48 mesh: unground, 24 mesh: 2TH 6900 rpm grind was selected as the best compromise between desirable propellant processing

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characteristics and lowered burning rate. The Arcane 40 variation containing this oxidizer blend was designated Arcane 40X; replacement of Brush "17-micron" with Brush "17 \pm 5 micron" beryllium changed the designation to Arcane 40CX.

Strand burning rate data for Arcane 40, Arcane 40X, and Arcane 40Y are contained in Figure II-1. These data are from laboratory batches of propellant which were burned uncured in straws.

Motor burning rates from 10-pound motor firings of Arcane 40, Arcane 40X, and Arcane 40CX are compared in Figure II-2. The average motor burning rate is lowered from 0.27 in/sec with Arcane 40 to 0.24 in/sec with Arcane 40X. Arcane 40CX and Arcane 40X do not differ significantly in motor burning rates, or, in fact, in any respect. (See the subsequent section on static firing data.)

Batch Chronology

Table III contains a detailed listing of all mixes made on this program from December, 1961, when the first 17-inch motor (17S-3) was cast containing Arcane 42, until August, 1962, when the final 17-inch motor (17SX-3) was cast containing Arcane 40CX. Also included in this table are a number of mixes made on other programs. Their inclusion helps to provide a more complete picture of the processing problems encountered.

After the casting of the first two 17-inch motors, which contained Arcane 42, efforts were directed toward producing the beryllium-containing propellant necessary for the tailoring program and subsequent evaluation firings. No cures were obtained in Arcane 40Y or Arcane 39Y, although most of these grains did "post-cure" after several weeks. In effect, the cure had been tremendously inhibited. The propellant grains would be subjected to a normal cure cycle (such that analogous aluminum-containing propellants would cure satisfactorily), without effecting satisfactory cures. Even doubling the cure cycle made no appreciable difference. The lack of cure was shown by the fact that the grains would pull away from the motor wall when the mandrel was removed.

In the case of the beryllium-containing propellants, the propellant slumped badly, sometimes immediately on removal of the mandrel, other times

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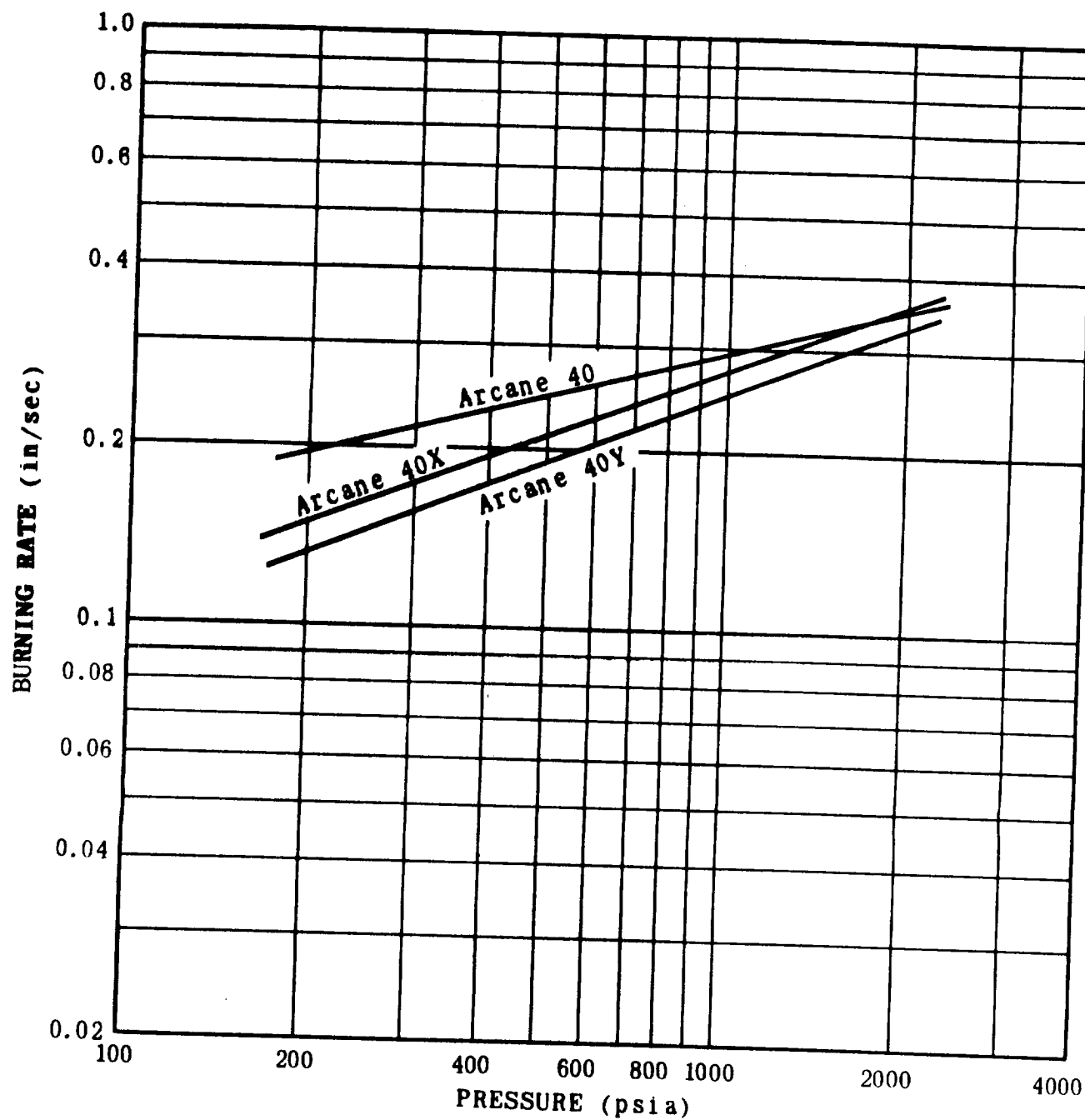


Figure II-1. Strand Burning Rates.

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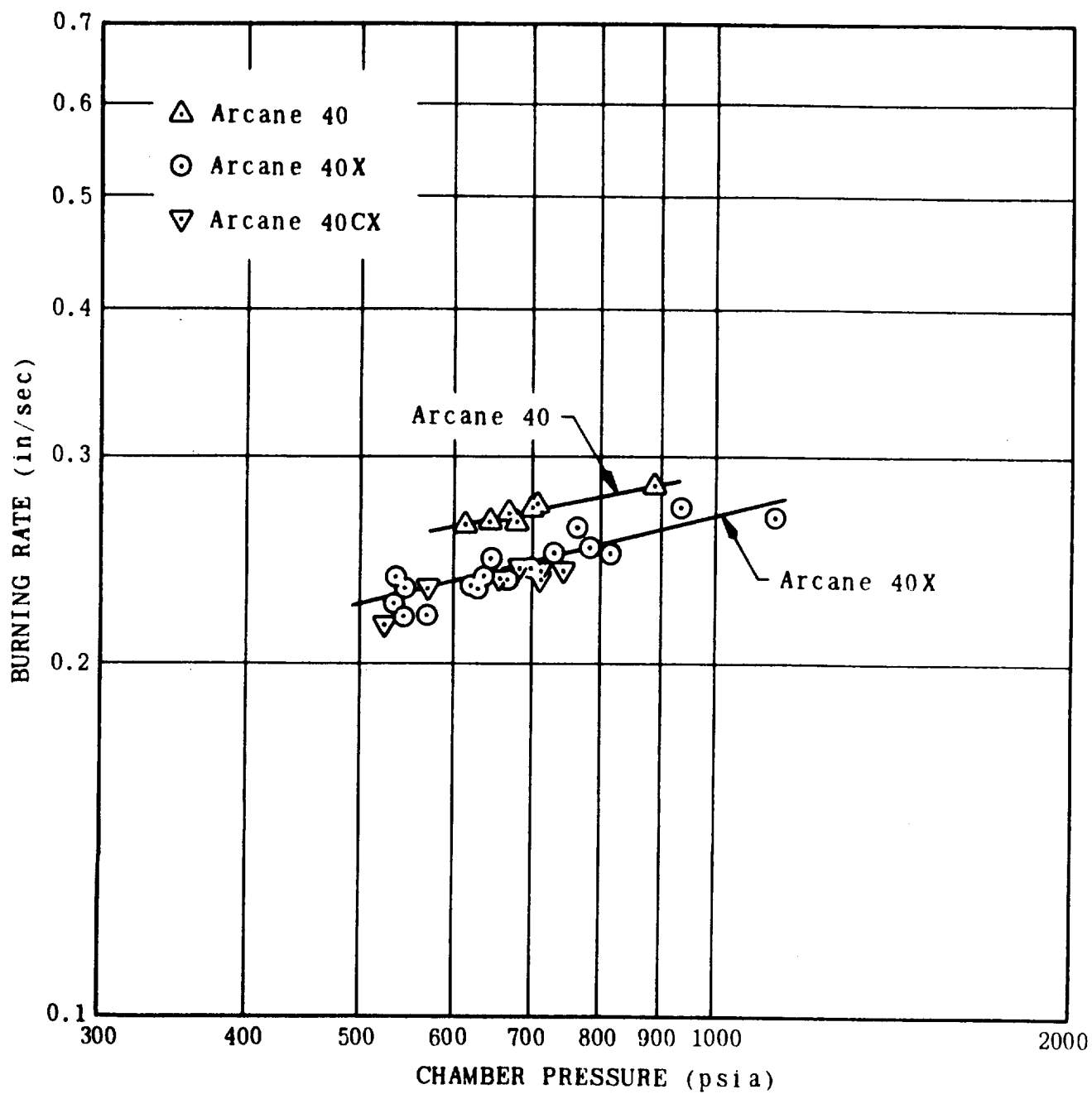


Figure II-2. Motor Burning Rates From 10-Pound Motor Firings.

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TABLE VII
CHRONOLOGICAL SUMMARY OF PROPELLANT MIXES

Date	Arms	Mix Number	Mix Size	Fuel Lot	Cure Time	Cure Temp.	WCD/MI ^b	Comments
12/15/61	42Y	137MA B	71,000g	Alcom 123	6.5 days	120	1.12/1.00	Cured
12/27/61	45Y	140E	12,000g	149	4 days	125	1.12/1.00	Not cured
					1 day	135		
12/29/61	40A	141E	11,000g	V-5505	3 days	135	1.12/1.00	Cured, but full of voids
					1 day	125		
1/2/62	40Z	142E	11,000g	149	2 days	135	1.12/1.00	Not cured
1/16/62	42	143MA B	71,000g	Alcom 123	6 days	125	1.11/1.00	Cured
1/19/62	42	150E	13,000g	Alcom 123	4 days	125	1.12/1.00	Cured
					1 day	135		
1/24/62	39A	152E	11,000g	A-052	4 days	135	1.07/1.00	Not cured
1/26/62	40Y	154E	12,000g	149	2 days	135	1.12/1.00	Not cured
1/31/62	40Y	157E	12,000g	149	5 days	135	1.08/1.00	Not cured
2/1/62	39Y	158E	11,000g	149	4 days	135	.97/1.00	Not cured
2/2/62	40Y	159E	11,000g	149	3 days	135	1.09/1.00	Not cured
2/6/62	40Y	160E	11,000g	149	4 days	135	1.04/1.00	Not cured
2/8/62	38	162E	10,000g	149	3 days	135	1.13/1.00	Not cured
2/9/62	42	163E	10,000g	Alcom 123	3 days	135	1.12/1.00	Cured
2/12/62	42	164E	10,000g	Alcom 123	3 days	125	1.12/1.00	Cured
2/13/62	42	166E	10,000g	Alcom 123	2.5 days	135	1.12/1.00	Cured
2/14/62	42	167E	10,000g	Alcom 123	2 days	125	1.12/1.00	Cured
2/15/62	42	168E	10,000g	Alcom 123	2 days	135	1.04/1.00	Cured
2/16/62	40Y	169E	11,000g	149	3 days	135	1.04/1.00	Not cured
2/19/62	42	170E	10,000g	Alcom 123	2 days	135	1.04/1.00	Cured
2/20/62	40Y	173E	6,000g	149	3 days	135	1.04/1.00	Not cured
2/20/62	40Y	174E	6,000g	23R	5 days	135	1.04/1.00	Poor
2/21/62	40Y	175E	6,000g	23R	1.5 days	135		Cured
2/22/62	40Y	176E	11,000g	23R	3.5 days	135	1.04/1.00	Poor
2/23/62	40Y	177E	6,000g	23P	4 days	135		Poor
2/26/62	40Y	178E	11,000g	23P	5 days	135		Poor

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TABLE III (cont'd)

Date	Arcane	Mix Number	Mix Size	Fuel Lot	Cure Time	Cure Temp.	NCO/ON ^b	Comments
2/27/62	30	179H	2,000g	23P	4.5 days	135		Poor
2/27/62	42Z	180H	2,000g	Alcon 123	4.5 days	135		Cured
2/28/62	40Y	181H	6,000g	23P	4 days	135		Poor
3/1/62	40	182H	2,000g	23P	3 days	135		Fair
3/2/62	40	184H	2,000g	149	3 days	135		Cured
3/5/62	40	185H	2,000g	149	Power failure		1.08/1.00	Not cured
3/5/62	40	186H	2,000g	149	Power failure		1.115/1.00	Not cured
3/5/62	40	187H	2,000g	149	Power failure			Not cured
3/8/62	40	188H	2,000g	23P	4 days	135	1.115/1.00	Poor
3/8/62	40	189H	2,000g	23P	4 days	135		Not cured
3/8/62	40	190H	2,000g	23P	4 days	135	1.08/1.00	Poor
3/9/62	40	191H	2,000g	23P	3 days	135		Poor
3/9/62	40	192H	2,000g	23P	3 days	135	1.08/1.00	Poor
3/9/62	42	193H	2,000g	Alcon 123	3 days	135		Cured
3/13/62	40	194H	6,000g	149	2 days	135		OK - .04 FeAA added
3/13/62	40	196H	6,000g	11	2 days	135		Cured - .04 FeAA added
3/14/62	40	197H	6,000g	11	2 days	135		Cured - .04 FeAA added
3/15/62	40	199H	6,000g	11	17 hours	135		Cured - .04 FeAA added
3/15/62	40	200H	6,000g	11	17 hours	135		Cured - .03 FeAA added. No difference
								Cured - .04 FeAA added
								No difference
								Cured - .03 FeAA added
3/16/62	40	201H	12,000g	11	17 hours	135		Cured - .03 FeAA added
3/20/62	40	203H	6,000g	11	24 hours	135		Cured - .03 FeAA added
3/21/62	40	205HA B	65,000g	11	24 hours	120		Cured - .03 FeAA added
3/22/62	40Y	206H	6,000g	11	5 days	135		Cured - .04 FeAA added
3/23/62	40Y	209H	6,000g	11	3 days	135		Cured - .04 FeAA added
3/23/62	40Y	210H	6,000g	11	2 days	135		Cured - .04 FeAA added
3/26/62	40W	211H	6,000g	11	2 days	135	1.10/1.00	Cured - .04 FeAA added better
					1 day	135		Cured - .04 FeAA added

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TABLE III (cont'd)

Date	Arcane	Mix Number	Mix Size	Fuel ^a Lot	Cure Time	Cure Temp.	MCQ/HR ^b	Comments
3/26/62	40X	212H	6,000g	11	1 day	135		Cured - .04 FeAA added
3/27/62	40X	213H	12,000g	11	2 days	125 135 ^c	1.10/1.00	Cured - .04 FeAA added
3/28/62	40X	214HA B	75,000g	11	8 days	125	1.10/1.00	Cured - .04 FeAA added
4/3/62	40X	217H	16,000g	14D	2 days	135		Cured - .04 FeAA added
4/4/62	40X	218H	17,000g	14D	2 days	135		Cured - .04 FeAA added
4/6/62	40X	220H	8,600g	400 mesh	3 days	135		1 poor, 1 fair, .04 FeAA added
4/9/62	39Z	221H	11,000g	14D	3 days	135		1 cured, 1 poor, .04 FeAA added
4/11/62	47	222H	12,000g	14D	2 days	135		Cured - .04 FeAA added
4/16/62	40X	225H	12,000g	14D	2 days	135		Cured - .04 FeAA added
4/19/62	40X	228H	12,000g	25	2 days	135		Cured, Porous, .04 FeAA added
4/20/62	39Z	229H	12,000g	25	2 days	135	1.10/1.00	Cured, Porous, .04 FeAA added
4/23/62	39AZ	230H	6,000g	A-042	2 days	135		Cured - .04 FeAA added
4/24/62	40X	231HA	6,000g	19S	3 days	135		Cured - .04 FeAA added
4/24/62	40X	231HB C	65,000g	14D 19S	3 days	135		Cured - .04 FeAA added
5/1/62	40X	236HA	10,000g	11	7 days	135		Cured - .04 FeAA added
5/2/62	40X	236HB C	69,000g	11 (72.4%) 14D (16.3%) 25 (11.3%)	3 days	125 135		1.10/1.00
5/11/62	42	247H	2,000g	Alcoa 123	3 days	125		Cured - .04 FeAA added
5/11/62	40X	248H	2,000g	25	3 days	125	1.10/1.00	Fair (Porous) .04 FeAA added
5/11/62	40X	249H	2,000g	25	3 days	125	1.10/1.00	Fair - .06 FeAA added (Porous)
5/12/62	40X	250H	2,000g	25	5 days	125	1.061/1.00	Fair (Porous) .06 FeAA added Slightly better cure
5/12/62	40X	251H	2,000g	25	5 days	125	1.082/1.00	Fair (Porous) .06 FeAA added
5/13/62	40X	252HA	10,000g	25	8 days	125	1.067/1.00	Fair (Porous) .06 FeAA added
5/14/62	40X	252HB C	69,000g	25	7 days	125	1.067/1.00	Fair - .06 FeAA added
5/23/62	40X	258H	2,000g	34	2 days	135	1.083/1.00	Poor - .06 FeAA added

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TABLE III (cont'd)

Date	Arcs	Mix Number	Mix Size	Feed ^a Lot	Cure Time	Cure Temp.	WCO/lot ^b	Comments
5/23/62	40X	255M	2,000g	34	2 days	135	1.083/1.00	Fair - .04 FehA added
5/23/62	40X	262M	2,000g	142	3 days	135		Fair - .04 FehA added
5/23/62	40X	263M	2,000g	34	3 days	135		Curd (Pecuma), .04 FehA added
5/30/62	40X	268M	2,000g	45	1 day	135		Not curd - .04 FehA added
5/30/62	40X	269M	2,000g	45	1 day	135	1.067/1.00	Not curd - .06 FehA added
5/30/62	40X	270M	2,000g	45	1 day	135		Fair - .08 FehA added
5/30/62	42	271M	2,000g	Alcon 123	1 day	135		Fair - .04 FehA added
5/31/62	40X	272M	2,000g	45	1 day	135		Fair - .08 FehA added
5/31/62	40X	273M	2,000g	45	1 day	135		Not curd - .04 FehA added
5/31/62	42	274M	2,000g	Alcon 123	1 day	135		Curd - .04 FehA added
6/21/62	42	291M	2,000g	Alcon 123	2 days	135		Fair - .04 FehA added
6/22/62	42	293M	2,000g	Alcon 123	1 day	135		Poor - .04 FehA added
6/22/62	42	294M	2,000g	Alcon 123	1 day	135	1.07/1.00	Poor - .04 FehA added
6/23/62	42	295M	2,000g	Alcon 123	1 day	135		Curd - .04 FehA added
6/23/62	42	296M	2,000g	Alcon 123	1 day	135	1.12/1.00	Curd - .03 FehA added
6/23/62	42	297M	2,000g	Alcon 123	1 day	135		Curd - .04 FehA added
6/24/62	42	298M	2,000g	Alcon 123	1 day	135		Curd - .04 FehA added
6/24/62	42	299M	2,000g	Alcon 123	1 day	135		Curd - .04 FehA added
6/26/62	42	301M	2,000g	Alcon 123	1 day	135		Curd - .04 FehA added
6/26/62	42	302M	2,000g	Alcon 123	1 day	135	1.12/1.00	Curd - .04 FehA added Slightly better
6/27/62	42	303M	65,000g	Alcon 123	5 days	135	1.12/1.00	Curd - .04 FehA added
7/25/62	42X	335M	2,000g	Alcon 123	5 days	135		Curd - .04 FehA added
7/25/62	40X	336M	2,000g	25	5 days	135		Fair - .04 FehA added
7/25/62	40X	337M	2,000g	IV-9	5 days	135		Curd - .04 FehA added
7/26/62	40X	338M	2,000g	25	4 days	135		Poor - .04 FehA added
7/26/62	40X	340M	2,000g	IV-9	4 days	135		Curd - .04 FehA added
8/3/62	40X	345M	6,000g	IV-9	3 days	135		Curd - .04 FehA added
8/6/62	40X	347M	11,000g	IV-9	3 days	135		Curd - .04 FehA added

TABLE III (cont'd)

Date	Arcane	Mix Number	Mix Size	Fuel ^a Lot	Cure Time	Cure Temp.	WCO/cm ^b	Comment:
8/8/62	40CX	349HA	6,000g	IV-9	2 days	135		Cured - .04 FeAA added
8/8/62	40CX	349HB	65,000g	IV-9	5 days	135		Cured - .04 FeAA added

^a Beryllium in all cases, except Alcos 123, which is an aluminum powder.

^b 1.05/.78/.11/.11 unless otherwise noted.

^c 2 grains, one at each temperature; no difference in cure.

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several hours later. Some grains actually pulled away completely and wound up as "puddles" of propellant in the bottom of the motor. Others came out intact on the mandrel and had to be pulled off. The unsatisfactory motors containing partially cured propellant were stored in a magazine area while awaiting disposal. Because of the contamination problem, disposition was scheduled similar to a static firing; unusable motors are burned in the static-firing tunnel. Because of a heavy firing schedule in the static-firing tunnel, some of these motors stayed in the magazine area awaiting disposal for periods up to two months. Examination of the propellant after storage showed that adequate cures had occurred in the majority of cases. The adequacy was, of course, judged solely by appearance; no physical properties could be measured. However, from the standpoint of hardness, tack-free surfaces, and flexibility of small cut samples, the propellant appeared to be cured.

The propellants which were formulated, Arcane 39Y and Arcane 40Y, contain 18 percent of binder. Their viscosity is quite high and the processing characteristics are marginal. These characteristics raised the question as to whether improper mixing was causing the curing difficulties. This theory was disproved by subsequent mixes which included some aluminum-containing Arcane 42 formulations and an Arcane 38 formulation. Arcane 38, which has 20 percent of binder, is considerably less viscous than either Arcane 39 or 40. In these mixes, the aluminum-containing formulations cured, and beryllium-containing formulations, whether having 18 or 20 percent binder, did not cure. Varying the isocyanate-to-hydroxyl ratio made no difference in the cure of the beryllium-containing propellants, although some differences in degree of cure were noted with the Arcane 42.

At this point, a consultation with JPL personnel pointed out the slight differences, previously mentioned, in the formulating of the JPL X-535 polyurethane binder. Mix 175H marked the introduction of JPL formulation and ingredients to the Atlantic Research work. It also was one of three mixes made from a small lot (23R) of Brush "17 \pm 5 micron" beryllium powder. A 10-pound motor suitable for static firing was cast from batch 175H. The grains made using beryllium from lot 23R, Atlantic Research Corporation ingredients

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and formulation were not as good as the grain made with JPL ingredients and formulation, but they were better than anything made previously. It appeared that more reliable cures could be obtained with polyurethane propellants containing "17 \pm 5 micron" beryllium than with those using ordinary "17-micron" beryllium or similar metal powders.

Using the JPL formulation, some improvement was noted in degree of cure, but no reproducible good cures were obtained in Arcane 38, Arcane 40, or Arcane 40Y. A mix of Arcane 42Y was made to investigate the possibility that the extra-large ammonium perchlorate might contain some impurity which interfered with the cure. This propellant cured satisfactorily.

The evidence overwhelmingly pointed to the beryllium powder as the source of trouble. As mentioned above, all grains cast from batch 11 of Brush "17-micron" beryllium cured satisfactorily. Mixes made from batch 14D and 19S, which were used concurrently with batch 11, cured but did not give as reliable results; in all cases it was found necessary to increase the catalyst content. Three subsequent lots of Brush "17-micron" beryllium, batches 25, 34, and 45, were used with very little success.

Available physical-property and burning-rate data for the batches of propellant used in the program are contained in Table IV. Usable test sheets for measuring physical properties and burning rates were not obtained in a great many of the cases, even when excellent cures were obtained in motors. Densities of propellants cast in 10-pound grains were calculated from grain measurements, and the results are reported in Table V.

Remedial Action

At this time processing studies were temporarily halted while other approaches were explored. Laboratory work demonstrated that the use of a pre-polymer based on polypropylene glycol and toluene diisocyanate would alleviate the curing problems. Evidently, the prereacting of part of the glycol and all the isocyanate provided sufficient deterrent to whatever kind of competitive reaction was taking place.

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BATCH PROPERTIES OF ARCANE PROPELLANTS

Formulation	Mix No.	Ballistic Data			Ultimate Stress (psi)	Tensile Properties		Young's Modulus (psi)
		Pressure (psia)	Burning Rate (in/sec)	Pressure Exponent		Yield Strain (%)	Ultimate Strain (%)	
Arcane 40A	141H	600	0.235	0.22	93		77	320
Arcane 40Z	142H	500	0.295	0.76	42		86	430
Arcane 38	162H	600	0.19	0.13	a			
Arcane 40Y	169H	600	0.25	0.30	a			
Arcane 40	197H	500	0.22	0.40	112	48	70	400
Arcane 40	199H	500	0.235	0.35	93	56	80	310
Arcane 40	200H	500	0.245	0.29	94	82	110	260
Arcane 40	201H	500	0.225	0.55	69	33	57	380
Arcane 40	203H	500	0.23	0.28	94	40	51	410
Arcane 40	205HA B	500	0.245	0.27	a			
Arcane 40X	212H	500	0.215	0.10	38	52	110	150
Arcane 40X	213H	500	0.21	0.38	165	38	61	480
Arcane 40X	214H-A	500	0.20	0.35	107	40	64	460
Arcane 40X	214H-B	500	0.23	0.34	113	44	68	480
Arcane 40Y	209H	500	0.21	0.13	a			
Arcane 40Y	211H	500	0.235	0.33	a			
Arcane 40X	217H	500	0.255	0.29	46	46	80	170
Arcane 40X	218H	a			60	37	54	260
Arcane 47	222H	500	0.27	0.30	a			
Arcane 40X	225H	500	0.24	0.20	109	57	83	410
Arcane 40X	228H	500	0.255	0.24	15	29	53	90
Arcane 39Z	229H	500	0.265	0.33	43	47	86	140

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TABLE IV (cont'd.)

Formulation	Mix No.	Pressure (psia)	Burning Rate (in/sec)	Pressure Exponent	Ultimate Stress (psi)	Yield Strain (%)	Ultimate Strain (%)	Young's Modulus (psi)
Arcane 4OX	231H-A	500	0.26	0.36	38	42	65	160
Arcane 4OX	231H-B	500	0.25	0.32	46	60	125	160
Arcane 4OX	231H-C	500	0.25	0.40	51	67	108	180
Arcane 4OX	236H-A	500	0.23	0.32	19	26	52	90
Arcane 4OX	252H-A	a			20	40	90	70
Arcane 4OX	252H-C	500	0.26	0.31	a			
Arcane 4OCX	34OH	500	0.20	0.30	30	36	96	110
Arcane 4OCX	345H	500	0.205	0.16	50	27	42	220
Arcane 4OCX	349H-A	500	0.19	0.26	40	33	55	200
Arcane 4OCX	349H-B	500	0.19	0.39	32	31	70	230
Arcane 4OCX	347H	500	0.20	0.24	a			

^a Sheets Not Cured

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The major problem in the use of prepolymers is the attendant increase in viscosity. In making a prepolymer of a glycol and an isocyanate, the resulting formulation is more viscous than the same propellant formulated from the individual ingredients without prereacting. When a prepolymer is used, some of the polymerization has occurred as "prepolymerization" and a more viscosity of the propellant increases further as the polymerization reaction continues. Since beryllium-containing polyurethane propellants with 18 percent of binder are already pressing the limits of satisfactory processing characteristics, the use of prepolymers was impossible without increasing binder content.

An alternate approach, the use of Brush "17 \pm 5 micron" beryllium, which had been proven to produce reliable curing characteristics, proved a better choice for this program, since it permitted completion of the work without changing the basic propellant formulation (Arcane 40). The use of prepolymerization would have required the formulation of a new propellant containing 20 percent or more of binder.

The prepolymerization approach was pursued successfully in another Atlantic Research program¹. Fifteen 10-pound motors loaded with beryllium-fuel, prepolymer-based Arcane propellants containing 20 percent of binder were processed, cured, and fired without difficulty. The physical properties and processing characteristics of these propellants proved satisfactory for large motor fabrication.

Conclusions

Polyurethane propellants fueled with beryllium can be successfully cast and cured if certain precautions are taken. The exact cause of non-reproducible curing is not known, but beryllium has been isolated as the source

¹ Atlantic Research Corporation, Development of High-Energy Solid Propellant Formulations, Contract No. AF 04(611)-8180.

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of the problem. Hence, tight control of the properties of the beryllium used is mandatory.

Beryllium particle size was shown to have a major effect on curing properties, and tight control of particle size is necessary. Propellants made from Brush "17 \pm 5 micron" beryllium powder cured satisfactorily; those made from the "17-micron" powder generally did not cure satisfactorily, but occasionally did. The fact that the division is not absolute indicates that particle size alone is not the critical factor. Table VI summarizes particle-size data on all lots of beryllium used. These data were provided by the suppliers.

There is no doubt that moisture is a factor in determining the degree of cure obtained in a polyurethane. Table VII gives the moisture contents of several lots of beryllium powder. However, no definite correlation between the moisture content of the beryllium powder and the curing characteristics could be established.

Metal powder analyses are also supplied by the manufacturers; these data are shown in Table VIII. The only variable that appears to correlate with degree of cure is carbon content, which was low in all batches that cured satisfactory.

The importance of carbon content is not completely clear. Smaller particle-size powders would be expected to contain more beryllium oxide and probably more organic contaminants, hence, more carbon (perhaps as beryllium carbide). Batch 11 was the only one with a weight average particle diameter (D_{50}) of less than 10 microns that cured satisfactorily, and it had the lowest carbon content of all batches used.

Ballistic Performance

Eighteen grains were fired in the tailoring program to evaluate propellants other than Arcane 40X (or Arcane 40CX). Twelve of these grains were Arcane 40 or variations, four were Arcane 39, and two were Arcane 47. The effect of increasing oxidation ratio (O_R) was explored with these three propellants, which have oxidation ratios of 1.0 (Arcane 40), 1.045 (Arcane 47),

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TABLE VI

PARTICLE SIZE ANALYSES OF BERYLLIUM POWDERS

Particle diameter, microns	By Sharpless Microanalyzer					By Coulter Counter					By ASTM E-20-51T					
	Batch Number	Batch Number	Batch Number	Batch Number	Batch Number	Batch Number	Batch Number	Batch Number	Batch Number	Batch Number	Batch Number	Batch Number				
	V6205	11	149	198	23F	23R	25	34	45	IV-9	11	25	34	45	AO43	A-052
30	93	100	100	100	100	96	100	100	100	99+	100	-	-	-	98	100
25	85	100	98	100	100	95	100	100	100	99+	99+	100	100	100	90	95
20	72	100	97	100	100	82	100	100	100	99	99	99+	99+	98	80	80
18	64	98	96	93	100	75	100	100	100	82	99	98	99	97	-	50
15	48	96	85	78	93	45	97	97	97	60	97	96	95	95	50	-
13	38	85	72	65	82	25	90	86	88	40	93	94	89	91	-	-
10	24	58	45	38	23	5.3	66	25	26	9.4	77	83	69	74	-	-
8	15	36	28	22	32	0	40	32	30	2.2	57	64	47	52	-	-
7	11	26	20	15	21	-	27	23	20	1.3	46	50	36	40	-	-
6	7.2	17	14	10	13	-	18	16	11	-	35	36	25	28	-	-
5	4.8	10	9.2	5	8	-	11	9.5	4.5	-	24	23	16	18	-	-
4	2.5	3.5	6.8	2.8	4.5	-	6	3.5	1	-	14	13.5	8.6	10	-	-

BERYLLIUM POWDERS FROM STATED PLANT

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TABLE VII
MOISTURE ANALYSES OF BERYLLIUM POWDERS
(OBTAINED WITH CEC MOISTURE ANALYZER)

<u>Batch Number</u>	<u>Percent Moisture</u>
11	0.19
14D	0.29
25	0.27
34	0.15
45	0.18
IV-9	0.14
Alcoa 123 (Aluminum)	0.04

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TABLE VIII
CHEMICAL ANALYSES OF BERYLLIUM POWDERS

Lot Number	A-043	A-052	V6505	11	14D	19S	23P	23R	25	34	45	IV-9	1194
Supplier ^a	Beryllco	Beryllco	Brush	Brush	Brush	Brush	Brush	Brush	Brush	Brush	Brush	Brush	General Astrometals
Ingredient, Weight Percent													
Be, assay	—	—	99.0	98.7	97.2	98.2	97.3	98.5	97.0	98.0	97.6	98.0	—
BeO	0.45	0.89	1.41	2.03	2.41	1.90	2.49	1.64	2.00	2.00	2.20	1.61	1.89
Fe	—	—	0.11	0.14	0.12	0.10	0.13	0.13	0.13	0.13	0.14	0.14	0.07
Al	—	—	0.11	0.11	0.10	0.08	0.10	0.06	0.30	0.21	0.26	0.13	<0.01
Si	—	—	0.03	0.07	0.04	0.05	0.06	0.04	0.17	0.12	0.11	0.08	<0.01
Mg	—	—	0.01	0.02	0.03	0.06	0.06	0.02	0.02	0.02	0.02	0.01	<0.001
C	—	—	0.11	0.09	0.30	0.24	0.25	0.11	0.39	0.26	0.33	0.12	0.02

^a Beryllco - The Beryllium Corporation, Hazelton, Pennsylvania
Brush - Brush Beryllium Corporation, Cleveland, Ohio
General Astrometals - General Astrometals Corporation, Yonkers, N.Y.

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and 1.09 (Arcane 39). No significant differences in specific-impulse efficiency were noted.

Thirty grains made in earlier portions of the program were not suitable for firing. These grains were burned out in the Atlantic Research tunnel.

Processing difficulties were encountered in making grains with either Berylco low-oxide beryllium or General Astrometals "5-micron" beryllium, and the data is too limited to correlate their performance with other materials.

The complete static-firing data are tabulated in Table IX.

Twenty-four 10-pound motors containing Arcane 40X (or Arcane 40CX) were static fired in the Atlantic Research static-firing tunnel, 14 at simulated altitude conditions and 10 under sea-level conditions. Included in these firings were three batch-check motors for JPL 17-inch motors and six batch-check motors for NOTS 100A or 100B 17-inch motors cast on another NASA program¹.

Table X provides a comparison between the data obtained at sea-level and at altitude conditions in these static firings. At the time these firings were made, the lower specific-impulse efficiency obtained in the altitude firings was believed to be an anomaly. Subsequent static firings on another program² showed that this was a real difference, which can be accounted for on the basis of thermodynamic calculations. Higher flame temperatures and higher mass-flow rate decrease the difference. An example of the effect of mass-flow rate is available in the data accumulated on this program. Arcane 40 (Table IX) is identical to Arcane 40X except for the ammonium perchlorate particle size and, hence, propellant burning rate and mass-flow rate. Seven firings of Arcane 40 and ten firings of Arcane 40X, all at sea level, had average specific-impulse efficiencies of 89.05 and 87.95 percent, respectively. The mass flow rate in the Arcane 40 firings was about 10 percent higher than that in the Arcane 40X firings.

¹ Atlantic Research Corporation, Contract NASI-1821, NASA Casting Program.

² Atlantic Research Corporation, Tailoring of High-Energy Propellant Ingredients, Contract AF 04(611)-8180.

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TABLE IX
BALLISTIC DATA SUMMARY
10-POUND GRAIN PROPELLANT EVALUATION
(CORRECTED DATA)

Prop Type	Test No.	Grain No.	Date Fired	Batch No.	Grain Wt. (lbs.)	Nozzle Ratio	P _{amb} (psia)	P _a (psia)	F _a (lbs.)	r (in/sec)	Measured Isp (lb-sec/lb)			I _{theo} (lb-sec/lb)	I _{eff} (%)		
											Analog Ch. 1	Digital Ch. 2	Average				
394Z	374	230H-1	5/9/62	Barylco A043	9.930	13.42	13.76	1073	845	0.328	-	247.21	250.02	248.61	284.86	87.27	
392	366	221H-1	5/8/62	14D	11.12	11.99	12.93	892	705	0.273	-	242.50	245.11	243.80	281.57	86.59	
392	369	229H-1	5/8/62	25	10.58	10.78	12.68	746	675	0.268	-	245.55	249.39	247.47	276.98	89.35	
392	370	229H-2	5/9/62	25	10.75	10.04	13.66	675	660	0.271	-	235.06	238.08	236.57	271.56	87.12	
40	298	195H-1	3/15/62	14B	11.10	5.777	15.82	700	695	0.271	-	237.71	237.69	237.70	266.31	89.26	
40	299	196H-1	3/15/62	11	11.02	5.517	12.80	613	670	0.263	-	238.34	238.26	238.30	268.02	88.91	
40	300	197H-1	3/16/62	11	11.10	5.896	15.16	686	670	0.264	-	236.99	237.00	236.99	267.09	88.73	
40	303	200H-1	5/7/62	11	10.93	12.15	18.10	890	780	0.284	-	234.81	237.33	236.07	270.38	87.31	
40	304	201H-1	3/19/62	11	11.00	5.753	12.83	649	685	0.267	-	240.58	240.68	240.63	269.56	89.27	
40	305	201H-2	3/19/62	11	10.95	5.699	13.06	642	675	0.264	-	241.35	241.32	241.33	268.41	89.91	
40	312	202H-2	3/28/62	11	10.95	4.152	13.77	705	685	0.274	-	236.75	235.77	236.26	262.53	89.99	
40Y	285	175H-1	2/27/62	23H	10.43	6.051	13.53	420	285	0.140	-	203.80	203.30	203.55	248.40	81.94	
40Y	310	209H-1	3/27/62	11	10.70	4.924	13.70	589	445	0.192	-	232.73	232.01	232.37	263.24	88.27	
40Y	309	210H-1	3/27/62	11	10.51	5.102	18.46	239	235	0.124	-	185.43	185.64	185.54	217.33	85.37	
40W	323	211H-1	4/3/62	11	10.96	4.354	16.65	533	450	0.180	234.87	236.60	236.86	-	236.11	255.01	92.59
40XX	348	220H-1	4/20/62	General Astronautal	9.978	9.940	13.89	440	435	0.221	-	217.17	219.10	218.13	254.50	85.71	
47	367	222H-1	5/8/62	14D	11.05	10.78	13.68	735	640	0.253	240.31	238.09	237.18	241.17	239.44	274.59	87.17
47	368	222H-2	5/8/62	14D	11.07	11.76	13.86	829	675	0.265	-	239.73	241.63	240.68	277.88	86.61	

TABLE X
COMPARISON OF SEA-LEVEL AND ALTITUDE PERFORMANCE OF ARCANÉ 40X

Firing No.	Arcane 40X Sea-Level Firings									
	311	324	325	338	339	387	388	389	575	611
P _c , psi	818	534	544	630	623	644	668	570	526	711
c	5.5	8.9	9.1	11.0	9.5	9.7	10.3	9.9	5.3	10.9
I _{sp} eff., %	89.23	89.32	87.48	86.99	85.95	87.69	88.25	88.23	89.01	87.30

Average = 87.95 percent

S. D. = 1.08

Firing No.	Arcane 40X Altitude Firings									
	350	351	352	353	355	356	357	358	359	541
P _c , psi	535	639	697	546	785	730	1126	935	762	742
c	39	47	49	40	50	49	68	59	50	59
I _{sp} eff., %	85.00	85.39	86.31	85.12	86.22	86.33	86.62	86.34	85.44	86.84

Average (all) = 86.35

S. D. (all) = 1.17

Average (-619 and 621) = 85.95

S. D. (-619 and 621) = 0.76

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Static firings are currently being conducted at Atlantic Research on an Air Force program¹. Several different beryllium-containing propellant compositions were evaluated in both sea level and altitude firings. One system in that program (System 2) evaluated beryllium-containing polyurethane propellants very similar to those used in this program. The most noticeable effect seen in the static firings of System 2 was that measured specific-impulse efficiencies at altitude conditions were lower than those at sea-level conditions. These results are in general agreement with those obtained on this program.

This poses the question of whether the difference in efficiency is due to difference in discharge pressures or in expansion ratios. Late in this program, three firings were conducted at expansion ratios of about 10 at simulated high altitude. The specific-impulse efficiencies measured in two of these firings (619 and 621, Table X) did not differ significantly from the efficiencies measured at sea level. (The third firing, 598, should be disregarded in this analysis, since it has a sharp break midway in the trace indicating some malfunction, such as case-bonding failure.)

These results indicate that expansion ratio is the parameter controlling the specific-impulse differences. The data obtained on the Air Force program show a difference of 1.6 percent in specific-impulse efficiency between the high and low expansion ratio used at constant propellant stoichiometry and constant mass-flow rate which is quite comparable to the two percent difference found in this program.

The following conclusions have been drawn based on the information from these two programs:

- (1) Super-cooling of liquid beryllium oxide probably occurs in the expansion process with these propellants, yielding losses in specific impulse that increase with expansion ratio.

¹ Atlantic Research Corporation, Development of High-Energy Solid Propellant Formulations, Contract No. AF 04(611)-8180.

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(2) Brush "17-micron", Brush "17 \pm 5 micron", and General Astrometals "400-mesh" beryllium powders yield equivalent performance in System 2 propellants under the conditions studied.

(3) In the range studied, specific-impulse efficiency is a strong function of mass-flow rate.

A more complete discussion of these effects, including information on double-base propellants, is available.¹

Substitution of Brush "17 \pm 5 micron" beryllium in Arcane 40CX for the Brush "17-micron" beryllium in Arcane 40X resulted in no change in the ballistic properties. In Table X, the last seven firings in chronological sequence (543 to 621) were made with Arcane 40CX.

Table XI contains the complete static firing data for the 24 firings of Arcane 40X. Attempts to correlate specific-impulse efficiencies with beryllium lot numbers have been unsuccessful. In sea-level firings, the specific-impulse efficiency from the static firing of two grains containing beryllium from lot 14D was about 2 percent lower than that obtained with the other lots tested, 11, 19S, and IV-9; one firing of a grain containing beryllium from batch 25 was about midway between the two groups. A similar comparison among the altitude firings (neglecting the underexpanded cases) gives the same ranking, but the effects of high expansion ratio somewhat overshadow the performance differences, i.e., the same differences exist but the magnitude of difference is decreased.

Two aluminum-containing control grains of Arcane 42 were static fired in the program. The results are summarized below:

Firing Number	242	248
Chamber pressure, psia	604	707
Average Thrust, lb	500	610

¹ Atlantic Research Corporation, Development of High-Energy Solid Propellant Formulations. Quarterly Progress Report No. 2, Contract AF 04(611)-8180, June 1 through September 30, 1962. CONFIDENTIAL.

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Firing Number	350 ^a	351 ^a	352 ^a	339	387	386	389	575	611
Grain Number	217H-1	217H-2	218H-1	218H-3	252H-1 ^e	236H-1 ^e	231H-1 ^f	351H-2 ^e	349H-1 ^f
Date Fired	4/24/62	4/25/62	4/26/62	4/12/62	5/22/62	5/22/62	5/22/62	9/7/62	9/27/62
Beryllium Lot Number	14D	14D	14D	14D	25	11	19S	IV-9	IV-9
Weight, Pounds	11.12	11.10	11.03	11.02	10.77	11.08	11.12	10.67	10.67
Average chamber pressure, psia	535	639	697	623	644	668	570	526	711
Average Exhaust pressure, psia	0.287	0.290	0.251	13.41	13.62	12.76	17.17	14.09	16.68
Expansion Ratio	38.6	47.2	48.6	9.54	9.67	10.34	9.91	5.26	10.94
Burning Rate, in/sec	0.236	0.237	0.241	0.233	0.246	0.236	0.220	0.216	0.236
Average thrust, pounds	725	745	760	580	625	610	530	620	570
Measured I _{sp} ^b , lb-sec/lb				-	-	-	225.8	232.8	234.4
Analog 1	286.8	290.5	296.3	-	-	-	225.1	233.0	234.4
Analog 2	287.3	293.2	296.5	230.7	238.7	242.7	226.7	232.6	234.7
Digital 1	287.2	290.6	295.4	233.9	236.2	241.0	224.7	231.7	234.0
Digital 2	287.9	294.7	297.4	232.3	237.4	241.8	225.6	232.5	234.4
Average	287.3	292.2	296.4	270.2	270.8	274.0	255.7	261.2	266.7
Theoretical I _{sp} ^c , lb-sec/lb	338.0	342.2	343.4	85.95	87.69	88.25	88.23	89.01	87.30
Average I _{sp} efficiency ^d , per cent	85.00	85.39	86.31	94.58	96.11	97.22	92.57	92.75	96.99
C* efficiency, per cent	90.64	88.13	92.48						

- a. Simulated altitude firings
b. Fifteen degree cone angle
c. Computed at the firing conditions, 0 degree cone
d. Measured I_{sp} times 100 divided by the theoretical
e. Batch check motors for another program (NASI-18)
f. Batch check motors for 17-inch spherical motors.

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I_{sp} measured ^a , lb-sec/lb	216.2	224.6
I_{sp} theoretical ^b , lb-sec/lb	240.3	252.6
I_{sp} efficiency, percent	90.0	88.9

^a 15-degree cone angle.

^b Computed at the firing conditions, 0-degree cone angle.

During the balance of the program, Arcane 42 mixes were used to check out cures, but no grains were cast. Ten-pound beryllium grains were used as batch check grains for the 150-pound motors.

CASTING OF 17-INCH MOTORS

Six 150-pound motors were cast during the program. The 17-inch spherical steel case were lined with General Tire and Rubber Company V-44 rubber asbestos by Frewitt Plastics Company, a subsidiary of Atlantic Research Corporation. The details of liner preparation prior to casting and the casting operation itself are included in Appendix C of this report.

In most cases, two mixes of propellant were made for each large motor casting. These two mixes were then cast into the motor simultaneously. Batch 303H (Arcane 42) and batch 349HB (Arcane 40CS), however, were each a single large mix. Pertinent data on the propellant batches are summarized below:

<u>Propellant</u>	<u>Batch Number</u>	<u>Date Cast</u>	<u>Cure</u>	
			<u>Temperature</u>	<u>Days</u>
Arcane 42	137H	12/15/61	120°F	6½
Arcane 42	148H	1/17/62	125	6
Arcane 40	209H	3/21/62	135	5
Arcane 40X	231H	4/25/62	135	7
Arcane 42	303H	7/10/62	135	5
Arcane 40CX	349H	8/10/62	135	5

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Physical property data and strand ballistic data determined from sheets cast from these mixes are presented in Table XII.

The grains were trimmed as described in Appendix B, and were subjected to radiographic examination. Two exposures were made perpendicular to the equator and at 90 degrees to each other. In addition, tangential views were obtained which helped to ascertain whether or not a good liner-to-propellant bond has been obtained. The heavy-walled steel case was heavier than desirable for obtaining good resolution, but the plates were fairly clear. These inspections showed no faults.

With each 17-inch motor cast, a 10-pound batch check motor was cast using the same ingredients. This motor was static fired at Atlantic Research Corporation's Pine Ridge plant prior to static firing of the large motor at the North Carolina facility. Firing data for these motors are presented separately in Table XIII.

CONCLUSIONS AND RECOMMENDATIONS

The processing of polyurethane propellants fueled with beryllium is not greatly different from similar operations carried out with aluminum-containing polyurethane propellants. The major difficulty is the reactivity of the metal. Whereas grades of aluminum currently employed in propellant manufacture are essentially inert toward conventional polyurethane ingredients, certain beryllium powders, or some constantly present impurity, inhibit polymerization of the polyurethane. This retards, or, in some cases, prevents, the cure of the propellant.

This difficulty can be overcome in two ways. One is the use of beryllium classified to remove the great majority of powder less than 10 microns in weight average particle diameter. Use of this powder greatly improves the reliability of cure. The second method involves prereacting part of the glycol and all of the isocyanate to form a prepolymer. This system has a very good reliability factor. The only drawback is the viscosity increase attendant with the

TABLE XII
PHYSICAL PROPERTIES AND BALLISTIC DATA FOR PROPELLANT BATCHES USED IN 150-POUND MOTORS

Batch	Cure		Ultimate Tensile (psi)	Elongation (percent)		Young's Modulus (psi)	Burning Rate (in/sec)	Pressure Exponent
	Time	Temp.		Yield	Ultimate			
137H-A	3 days	125°F	81	—	80	400	0.23	0.24
137H-A	3 days	125°F	—	—	—	—	—	—
	+2 days	140°F	130	—	90	300	—	—
137H-B	3 days	125°F	65	—	130	300	0.225	0.24
137H-B	3 days	125°F	—	—	—	—	—	—
	+2 days	140°F	105	—	90	400	—	—
148H-A	2 days	140°F	74	48	80	300	—	—
148H-A	4 days	140°F	140	56	70	600	—	—
148H-A	2 days	125°F	67	50	79	350	0.25	0.26
148H-B	3 days	125°F	90	52	79	500	—	—
148H-B	4 days	125°F	115	56	86	500	—	—
148H-B	5 days	125°F	148	56	77	600	—	—
148H-B	6 days	125°F	116	50	83	400	0.245	0.30
148H-B	2 days	140°F	107	46	70	700	—	—
148H-B	4 days	140°F	135	64	85	600	—	—
205H	SHEETS DID NOT CURE							
231H-A	—	—	—	—	—	—	0.245	0.27
231H-B	—	—	—	—	—	—	0.26	0.36
231H-C	—	—	—	—	—	—	0.25	0.32
303H	2 days	140°F	93	41	60	360	0.25	0.40
303H	4 days	140°F	109	38	60	425	0.265 ^a	0.46
303H	5 days	140°F	98	44	60	420	—	—
303H	5 ^b days	140°F	86	32	50	385	—	—
349H	SHEETS DID NOT CURE							
							0.19	0.26

^aAt 700 psi.^bPlus four days at ambient.

TABLE XIII

TEN-POUND BATCH CHECK MOTOR DATA

Grain Number	205H-2	214H-1	231H-1	236H-1	252H-1	349H-1
Date Fired	3/28/62	4/27/62	5/22/62	5/22/62	5/22/62	9/27/62
Average P_c , psia	705	730	570	668	644	711
Average P_c , psia	13.77	0.301	17.17	12.76	13.62	16.68
Expansion Ratio	4.15	49.0	9.91	10.34	9.67	10.94
R_b , in/sec	0.274	0.248	0.220	0.236	0.246	0.236
Average I_{sp} , lb-sec/lb	236.3	296.3	225.6	241.8	237.4	234.4
Theoretical I_{sp} at Firing Conditions, lb-sec/lb	262.6	343.2	255.7	274.0	270.8	266.7
I_{sp} Efficiency, per cent	89.99	86.33	88.23	88.25	87.69	87.30

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use of a prepolymer which permits propellant processing at no less than 20 percent binder. From a performance standpoint, however, there is little difference. A slightly higher maximum theoretical specific impulse (283.1 vs. 282.4) is calculated at 20 percent binder than at 18 percent binder. This offsets the very slight difference in density at practical mass-to-volume ratios.

The combination of these two processes results in a propellant system which is quite reliable. This has been quite convincingly demonstrated in other programs.

The static-firing data, although originally disappointing in character, paved the way for significant advances in the understanding of the parameters affecting propellant performance. Subsequent programs have shown that the effects of mass-flow rate and nozzle expansion ratio are predominant in determining the specific-impulse efficiency.

Any future work on high-performance propellants for large motors should utilize the experience gained in this program and concentrate on the prepolymer-based propellant fueled with the specially classified beryllium powder. Motor designs such that maximum mass-flow rates are possible will undoubtedly result in significant performance enhancement.

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III. MOTOR COMPONENTS

INTRODUCTION

The primary objective of the program was the design of a 36-inch-diameter spherical rocket motor having as high a propellant mass fraction as could be achieved. The components designed for this motor were to be evaluated by stress analysis and by the testing of 17-inch-diameter subscale motors. Two types of subscale motors were designed for test: a heavy-walled steel motor and a titanium motor of flightweight wall thickness. The heavy-walled motors were to be static fired to evaluate motor assembly techniques as well as the design characteristics of the major components including the propellant grain, the nozzle and nozzle-retention system, the case insulation-liner material, and the igniter. The 17-inch titanium motors were to be used for hydrostatic burst tests and for firings to prove the prototype motor case and the final motor assembly design and procedure. Subscale nozzle components were to be subjected to char-rate and hydrostatic proof pressure tests.

Propellant development is discussed in the preceding section of this report; the design and development of the other major motor components are discussed below.

MOTOR CASE

The designs of the titanium and heavy-walled steel cases for the subscale, 17-inch spherical motors are shown in Figure III-1. Both cases are formed by welding a spherical segment onto a hemispherical shell having an internal diameter of slightly greater than 17 inches. Each has an 8-inch-diameter opening at the after end to allow the nozzle to be completely submerged within the case. The steel unit, fabricated from AISI 4130 steel, has a wall thickness of 0.075 inch. The titanium case consists of 6AL-4V

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titanium alloy and has a wall thickness of only 0.03 inch. Welded into the after end of each case is a nozzle retention flange, drilled to accommodate 24 equally spaced studs. To measure chamber pressure in static firings, two diametrically opposed studs are removed and replaced with pressure taps.

Six steel and seven titanium cases were fabricated early in the program. All six of the heavy-walled steel motors were fired in static test; the firing of titanium motors was prohibited by the depletion of contractual fund allocations. The steel cases all successfully withstood a hydrostatic proof pressure of 900 psi, applied for 3 minutes, prior to being loaded for test. No case problems were experienced in the six static firings.

One of the subscale titanium cases was subjected to a hydrostatic burst pressure test at the Wyle Laboratories, El Segundo, California. This test was terminated prior to failure when the bolts holding the nozzle pressure plate on the case failed at approximately 1,150 psig. At this pressure, a maximum case stress of 169,500 psi was recorded at the extreme after end of the case. At the design motor proof pressure of 1,000 psi, a maximum stress of 138,700 psi was recorded at a location 1.5 inches from the after plane. Since these stresses corresponded to those predicted by theoretical calculations, it was concluded that the titanium case was suitable for its intended application. A detailed report covering the results of this pressure test, MED Report No. SR 205, was submitted to the Jet Propulsion Laboratories in May 1962.

A 36-inch-diameter titanium spherical motor of minimum weight was designed, and a stress analysis was completed in August 1962. The design criteria for the case are listed below:

Minimum tensile strength at room temperature	170,000 psi
Minimum yield strength at room temperature	165,000 psi
Minimum yield strength at 150°F for short term duration.....	155,000 psi
Membrane design stress.....	150,000 psi
Combined stress limit	155,000 psi
Design proof pressure	1,000 psi
Peak operating pressure	800 psi
Minimum margin of safety	0.25

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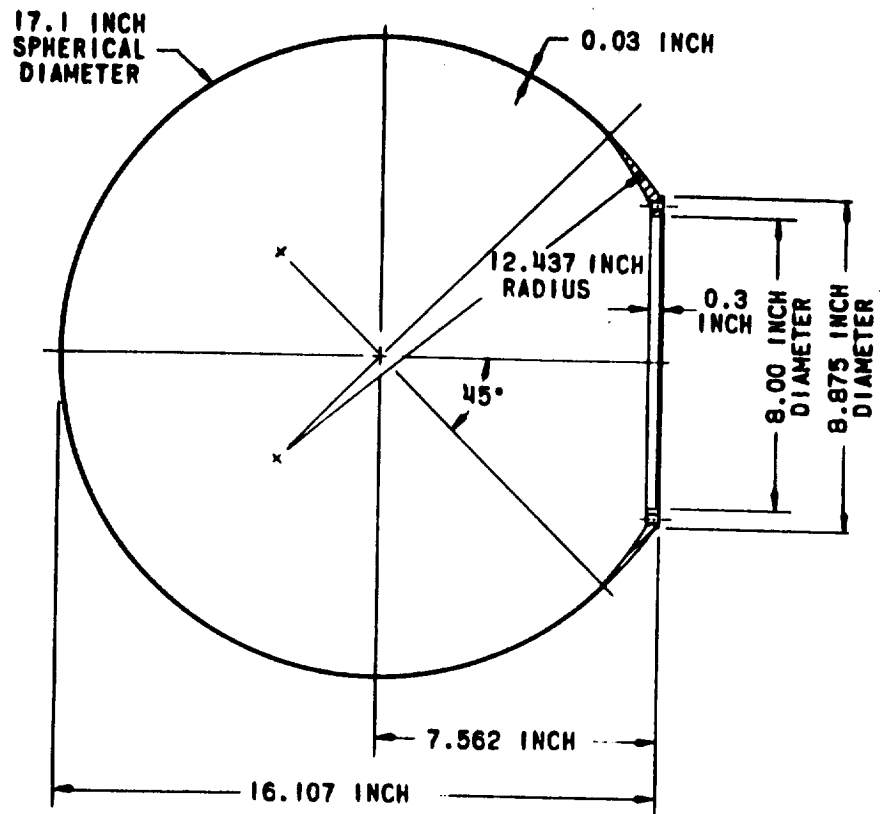


FIGURE 2. TITANIUM MOTOR CASE

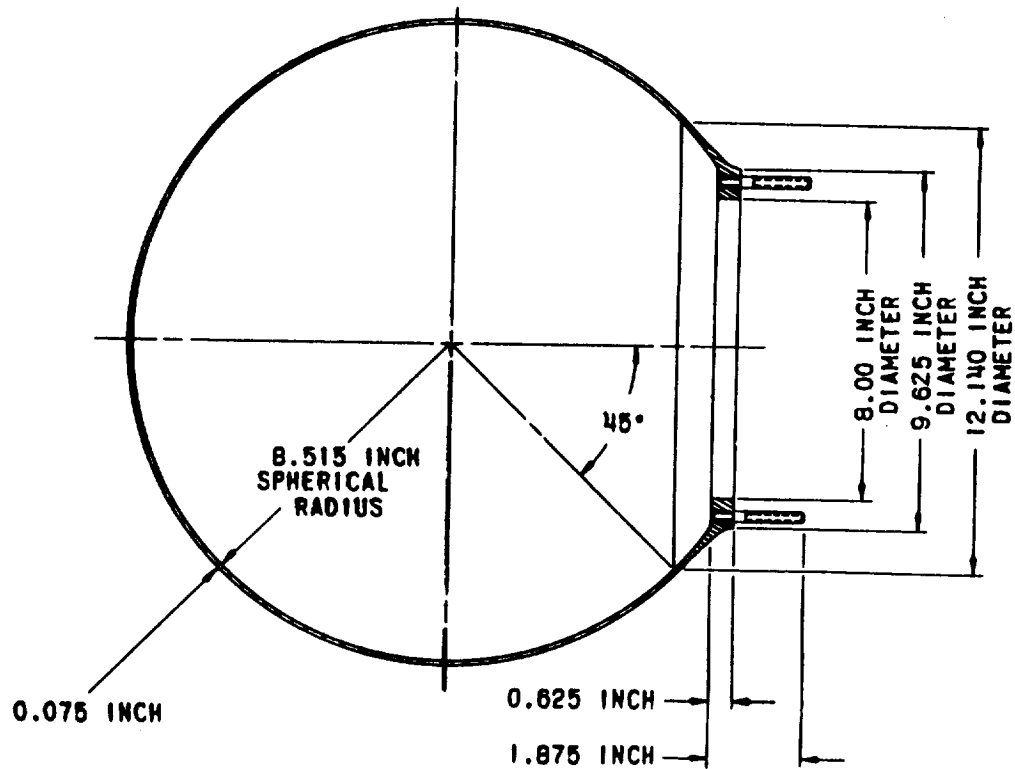


Figure III-1. Heavy-Wall Steel Motor Case.

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Data used in the stress analysis were derived from the design, fabrication, and hydrostatic test of the 17-inch titanium spherical case. Details of the analysis are shown in pages 2 through 13 of MED Report No. SR 207, included as Appendix D.

CASE INSULATION MATERIAL

The insulation liner used in the six heavy-walled steel, 17-inch spherical motors was fabricated from Gen-Gard V-44 asbestos rubber, a product of the General Tire and Rubber Company. This liner served to protect the case from the extreme combustion temperatures associated with the Arcane propellants.

Prior to application of the insulation, the internal surface of the motor case was sanded with 50-grit sandpaper to remove rust and foreign matter. The surface was then thoroughly cleaned with methyl ethyl ketone. A coat of Bloomingdale Rubber Company's No. 305 adhesive was applied to the prepared surface and cured for 10 minutes at 300°F. With the case still hot, the surface was first coated with V-44 rubber emulsified with methyl ethyl ketone and then covered with strips of the V-44 insulation. A rubber bladder was then inserted into the case and pressurized to 100 psi to ensure intimate contact between the strips and the case surface. This assembly was then heated for 1 hour at 300°F to vulcanize the rubber sheets. After this period, the assembly was allowed to cool to room temperature, and the rubber bladder was removed. The lined case was then visually inspected for insulation flaws or other defects. The insulation thickness and the total insulated surface were varied in the six heavy-walled cases to determine the optimum insulation thickness and area required to protect the case during motor operation.

NOZZLE

The nozzle was designed to be completely submerged within the motor case and to be retained by attachments fastened to the motor case by means of the studs in the case flange. The essential parts of the nozzle are a

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molded asbestos-phenolic cone and a graphite nozzle-throat insert. The preliminary nozzle design for the prototype titanium motor is shown in Figure III-2. The nozzle in this design is contoured and incorporates a diffuser section affording a greater nozzle expansion ratio. The material selected for the phenolic cone was Raybestos-Manhattan 150 RPD, which has an ultimate compression stress of 26,000 psi and an ultimate tensile stress of 6850 psi. The stress analysis for the minimum weight nozzle designed for the full-scale, 36-inch-diameter motor is included in pages 14 through 25 of MED Report No. SR 207, attached as Appendix D.

Early in the program, a test was devised to obtain char-rate data with a heavy test nozzle made from the 150 RPD material. This test failed when the special test motor over-pressurized and sheared the retaining rings holding the nozzle adapter in place. The test, however, appeared to have proven the structural integrity of the nozzle and nozzle-retention system since the failure occurred at a pressure of 1,500psi, a value almost twice that of the expected peak motor operating pressure. A nozzle system of the same design was therefore consigned for use in static firing 17S-1.

The nozzle used in 17S-1 consisted of a 150 RPD asbestos-phenolic cone with an integral retention flange and a graphite liner bonded to the entire internal surface of the cone. The only significant difference between this system and the one employed in the char-rate test was in the retention rings placed between the stud nuts and the nozzle flange. In the char-rate test, these rings consisted of a 1/2-inch phenolic ring and a 1/4-inch steel ring, both of which covered the entire flange surface to the graphite liner interface. In firing 17S-1, however, only a 1/16-inch steel ring was used to cover the area immediately around the studs. The firing resulted in nozzle expulsion after 1 second of burning at an approximate chamber pressure of 420 psi. A similar failure occurred upon ignition in a subsequent firing, test 17SX-2.

After the second expulsion of a nozzle during static test, two 150 RPD asbestos-phenolic cones were hydrostatically pressure tested. At approximately 400 psi, these nozzles failed in tension at a location opposite

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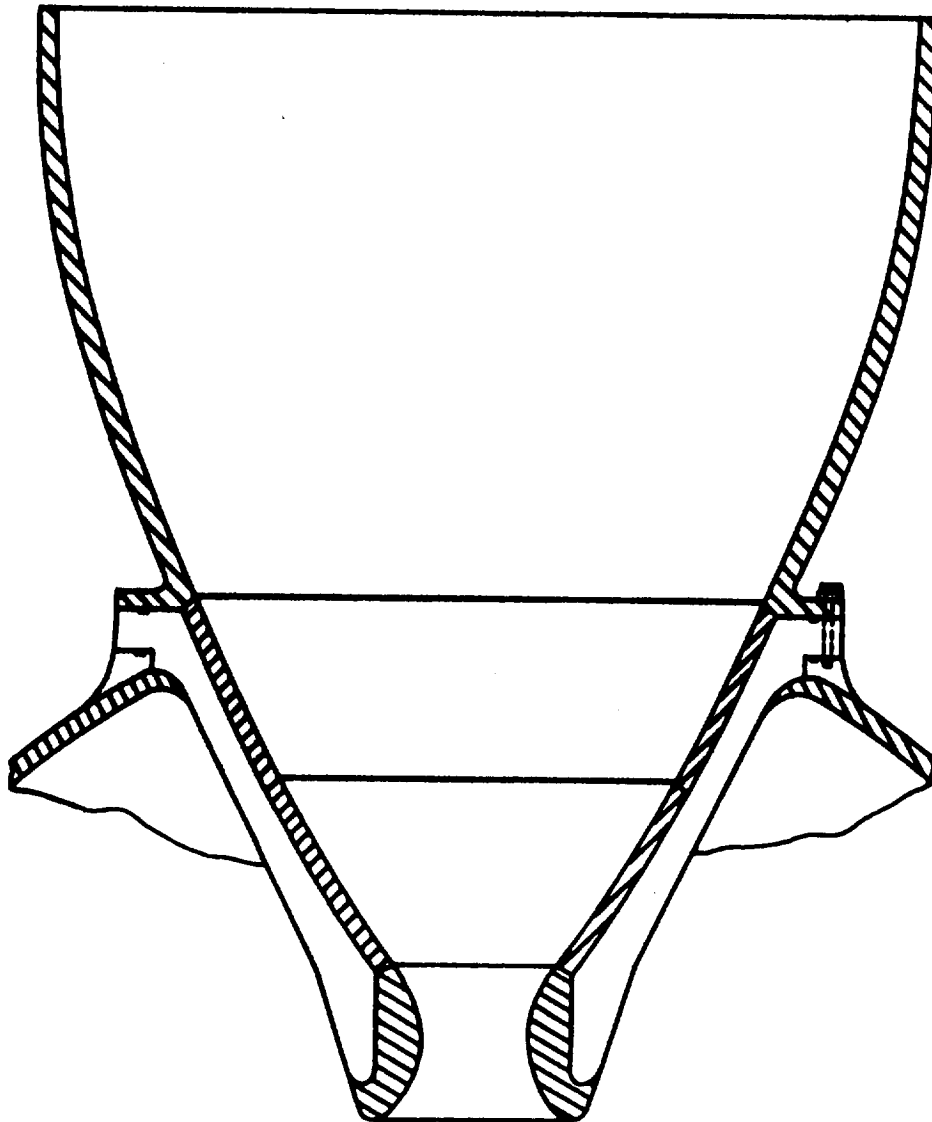


Figure III-2. Preliminary Nozzle Design for
Solid-Propellant Spherical Motor.

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the O-ring groove, about 1 inch forward of the nozzle exit plane. The resultant cracks propagated completely around the internal conical surface of the nozzles as shown in Figures III-3 and III-4.

It was concluded from the pressure tests that the nozzles in firings 17S-1 and 17SX-2 had also failed under tensile pressure. The thicker plates used in the char-rate test had apparently prevented a similar failure from occurring in the char-rate test. Hence, the nozzle was redesigned to incorporate a heavy steel retaining ring in place of the integral flange on the asbestos-phenolic cone. A nozzle of this design was proof tested under a hydrostatic pressure of 900 psi applied for 1 minute; there was no evidence of damage to either the nozzle or its retention system. Figures depicting the various design stages of the nozzle for the 17-inch subscale motors are included in Section IV of this report.

As a result of the char thicknesses and erosion measured in expended motors after firing, it became evident that the wall thickness of the 150 RPD nozzle cone could be reduced. Hence, a hydrostatic pressure test was performed on a lightweight nozzle cone with an aluminum retaining ring. This cone was held in place in the pressure vessel as shown in Figure III-5. At a pressure of 750 psi, the nozzle failed at the O-ring groove as the result of a bending moment induced at the outer edge of the after end of the nozzle exit cone. This bending moment was produced by the unexpected yielding and deformation of the aluminum retaining ring and the consequent shifting of the load to the outer periphery of the nozzle. A photograph of the failed cone is presented in Figure III-6. Subsequent analysis by the discontinuity theory clearly predicted a similar failure under actual loading conditions.

NOZZLE CONTOUR STUDY FOR 36-INCH SPHERICAL MOTOR

Five optimum nozzle contours were designed according to the method of G.V.R. Rao¹. The expansion process was assumed to occur with temperature and

¹Rao, G.V.R., "Exhaust Nozzle Contour for Optimum Thrust," Jet Prop. Vol. 28, No. 6, June 1958, pp. 377-382.

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velocity equilibrium existing between the liquid and gaseous phases in the combustion products. This assumption was necessary in that the application of the design method is valid only for an isentropic flow process. A ratio of specific heats representative of the two-phase mixture was used in the design of the contours. Fortunately, the geometry of the optimum contours is not sensitive to the choice of this ratio¹. All of the optimum contours were designed for vacuum operation. The performance of the five contours was evaluated assuming isentropic flow and a gas composition frozen at chamber conditions. The thrust produced by each of the nozzles was determined from the nozzle-flow field as generated by the method of characteristics. The thrust thus obtained was divided by the one-dimensional thrust obtained under the same assumptions and at the same area ratio. This quotient is then a measure of the thrust momentum lost to the radial component of the flow velocity. Also inferred in the application of this efficiency to the two-phase flow is the assumption that the particle radial velocity lags in the actual flow are negligible at the nozzle exit and at the two-phase gas streamlines are coincident with the computed no-lags streamlines. A paper by Winer and Morey² lends some validity to this assumption. Figure III-7 shows the results of this calculation.

The loss in delivered performance due to temperature and velocity differences between the two phases present in the exhaust gases was obtained by computing the one-dimensional two-phase flow field. Several published works^{3,4} are available presenting the derivation of the equations describing the isentropic expansion of the two-phase flow. The integration of these equations is a straightforward process on a high-speed electronic computer. The specific impulse figure thus obtained was divided by the one-dimensional no-lags impulse

¹Rao, G.V.R., "Optimum Thrust Performance of Contoured Nozzles." Paper presented at Liquid Propellant Information Agency Conference, December 1959.

²Winer, Richard, and L. Morey, "Nozzle Design for Solid Propellant Rockets." Presented at the Solid Propellant Rocket Research Conference, Princeton University, Princeton, N.J., January 28-29, 1960.

³Kliegel, J.R., "One Dimensional Flow of a Gas Particle System." Institute of Aeronautical Sciences, Preprint No. 60-5, January 25, 1960.

⁴Bailey, W.S., "Gas Particle Flow in an Axisymmetric Nozzle," American Rocket Society Journal. June 1961, pp. 793-798.

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Figure III-3. Hydrostatic Pressure Test Setup After Test No. 1.

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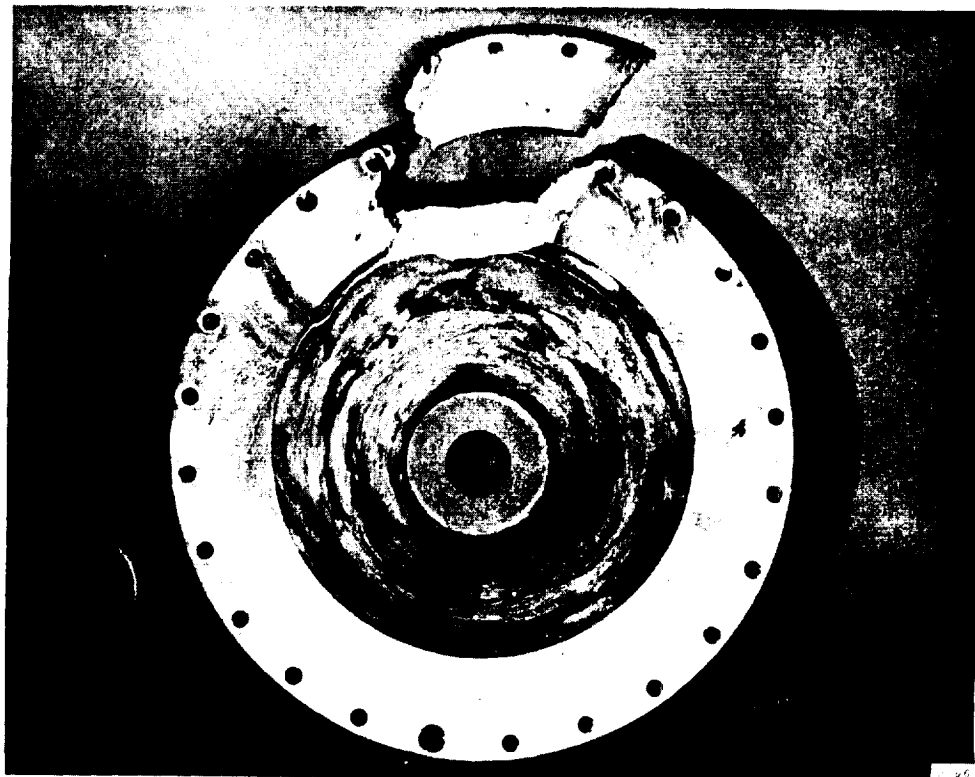
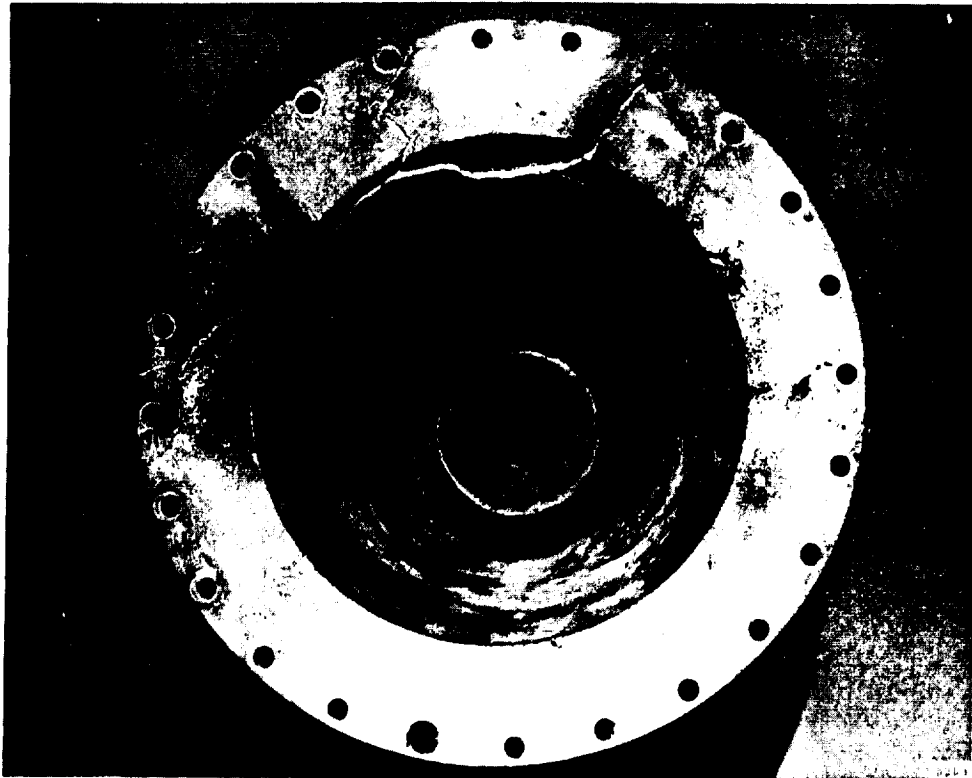


Figure III-4. Nozzle After Hydrostatic Pressure Test No. 2

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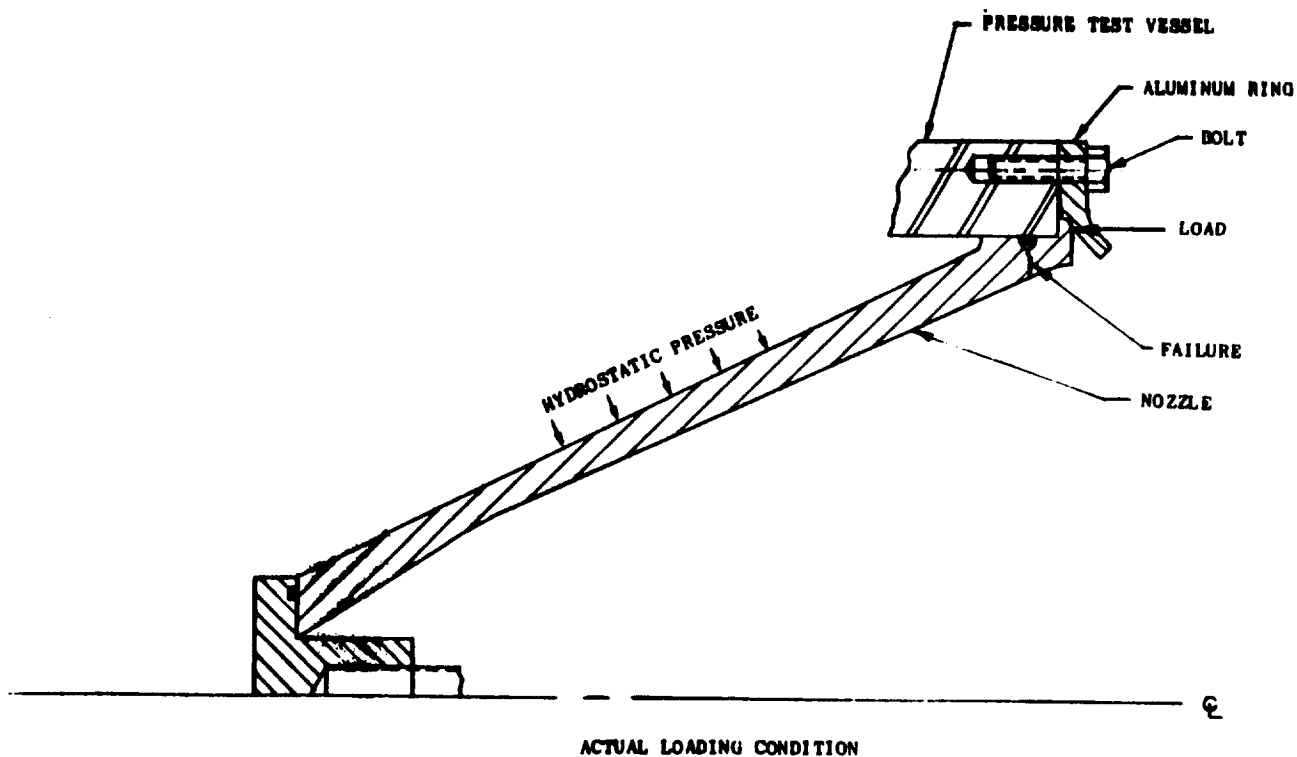
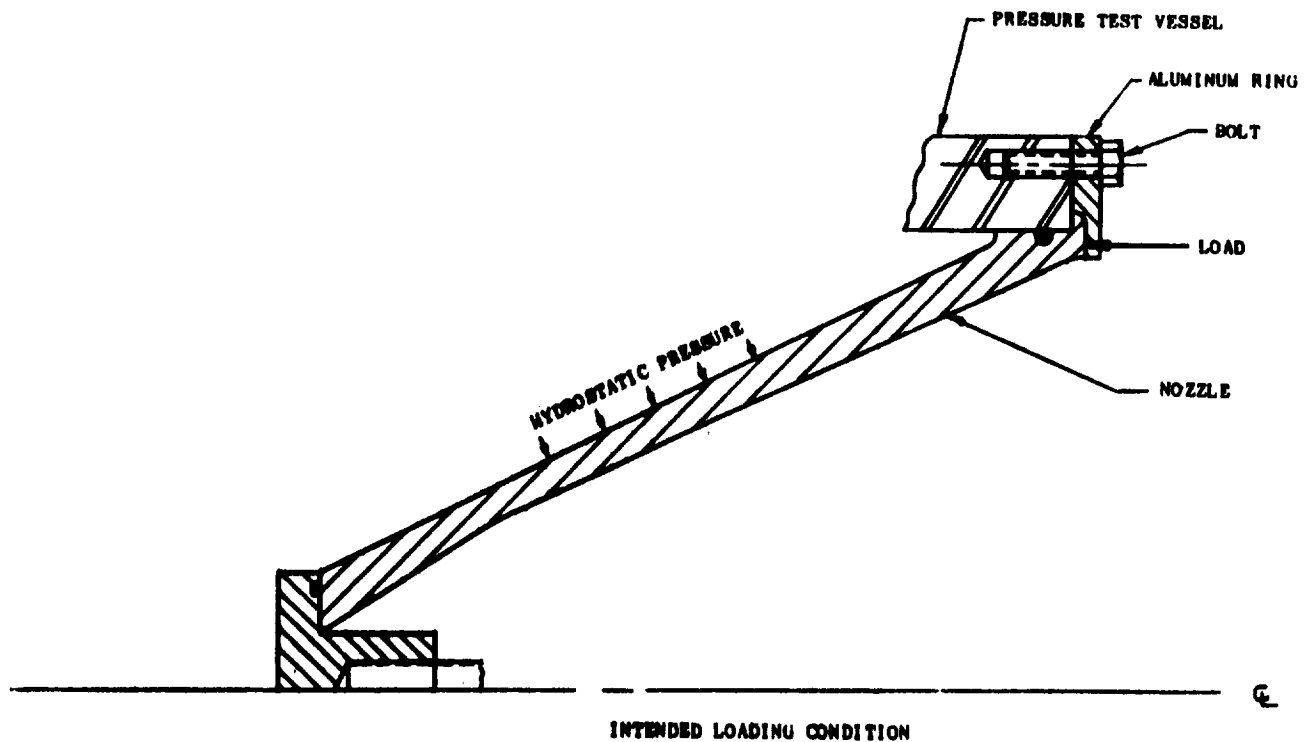


Figure III-5. Hydrostatic Pressure Test of Nozzle Cone and Retaining Ring for 17-inch Spherical Motor.

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Figure III-6. Molded 150 RPD Nozzle Cone Tested to Failure
Under Hydrostatic Pressure of 750 psi.

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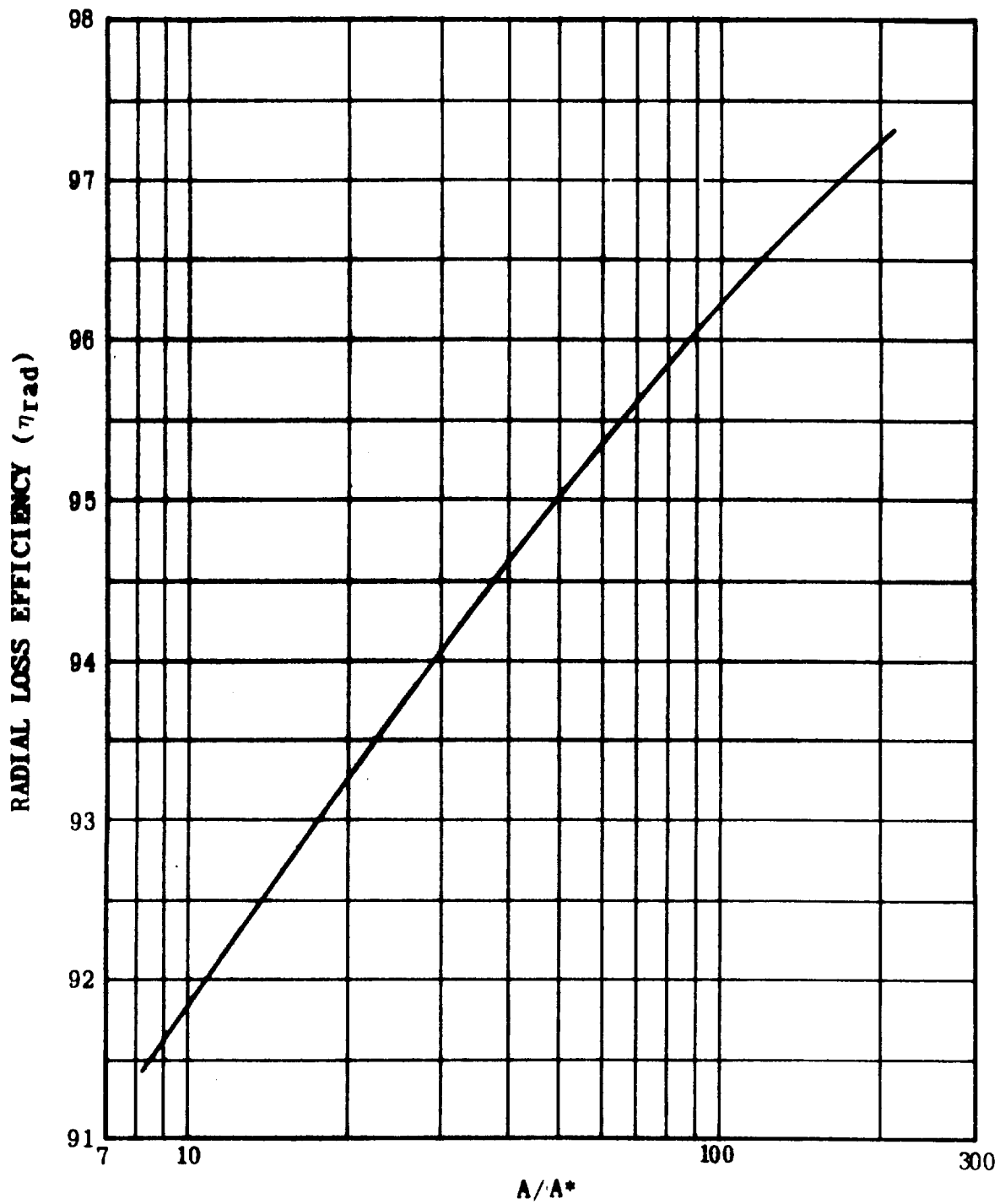


Figure III-7. Rao Nozzle Radial Loss Characteristics.

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obtained previously, thus providing a measure of the lag losses. As pointed out by Kliegel¹ the one-dimensional calculation leads to results which are quantitatively as good as the results from the more difficult axisymmetric calculation. Figure III-8 shows the variation of lag efficiency with area ratio for the optimum contours.

Figure III-9 shows the calculated ideal impulse and the expected delivered impulse. The ideal impulse is computed assuming shifting chemical equilibrium, one-dimensional flow, and no lags and represents the maximum impulse obtainable from the propellant formulation. An assumed combustion efficiency of 95 percent and a heat and momentum boundary layer loss of 1/2 percent, along with the lag and radial momentum losses, constitute the major losses which combine to provide an expected over-all efficiency. The over-all efficiency is applied to the ideal specific impulse to give the expected delivered specific impulse shown in Figure III-9.

Figure III-10 shows the variation of nozzle structure weight with nozzle length for the optimum contours. The structure weights were obtained by laying the five contours on an assembly drawing of the motor. No attempt was made to optimize the nozzle retention structure with respect to flange diameter. This penalizes the shorter nozzles to some degree because the area varies more slowly with length in the shorter nozzles, thus allowing the short nozzles to penetrate more deeply into the motor. The weight of the internal nozzle structure is necessarily heavier than the external structure since the exhaust gases contact both the inner and outer surfaces of the nozzle.

Figure III-11 shows the calculated payload and total up-stage weight variation with nozzle length. These calculations include all motor hardware in the final configuration but do not include interstage coupling and aerodynamic fairings. These weights would in any case be charged to the payload of the previous stage assuming they are jettisoned or left behind upon stage ignition.

The conditions for the optimization are as follows:

Velocity increment	8,430 ft/sec
Maximum total up-stage weight	2,500 pounds

¹Kliegel, J.R., and Ganz Nickerson, "Flow of Gas-Particle Mixtures in Axially Symmetric Nozzles." American Rocket Society Conference on Propellants and Combustion and Signal Rockets, Palm Beach, April 26, 1961.

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Inert parts weight (less nozzle)

Case	44.4 pounds
Studs	0.43 pound
Insulation	11.00 pounds
Attachment lugs	15.75 pounds
Fuel weight	1,280 pounds

It is evident from Figure III-11 that the optimum nozzle length lies outside of the stage space envelope. A nozzle having a length of approximately 25 inches was chosen for the final design. This choice results in a payload capability of 787 pounds and a stage gross weight of 2,178 pounds. Since the resulting total up-stage weight is less than the maximum allowable, it may be advisable to extend the stage envelope by adding additional interstage structure. This decision depends on a performance evaluation of the entire system; however, the information provided can be used in making such a decision.

Figure III-12 shows the evaluated performance of a conical contour of the same length as the chosen Rao contour. Several nozzle half-angles were used in the calculations to determine the best performing conical contour. As can be seen, the Rao contour outperforms the best conical nozzle by a little more than 2 percent. This difference is greater than that reported in similar investigations^{1,2}, and is probably optimistic by 0.5 percent.

ATTACHMENT LUG FOR THE 36-INCH SPHERICAL MOTOR

A subscale attachment lug was tested to obtain the information required to determine the contact area for the lug to be used on the 36-inch spherical motor. The lug dimension scaling ratio was 2:1, and the load scaling ratio was 4:1.

¹Winer, Richard and L. Morey. "Nozzle Design for Solid Propellant Rockets". Presented at the Solid Propellant Rocket Research Conference, Princeton University, Princeton, N.J., Jan. 28-29, 1960.

²Bloomer, Harry E., et al., "Experimental Study of Effects of Geometric Variables on Performance of Contoured Rocket Engine Exhaust Nozzles." NASA TN-D1181, January 1962.

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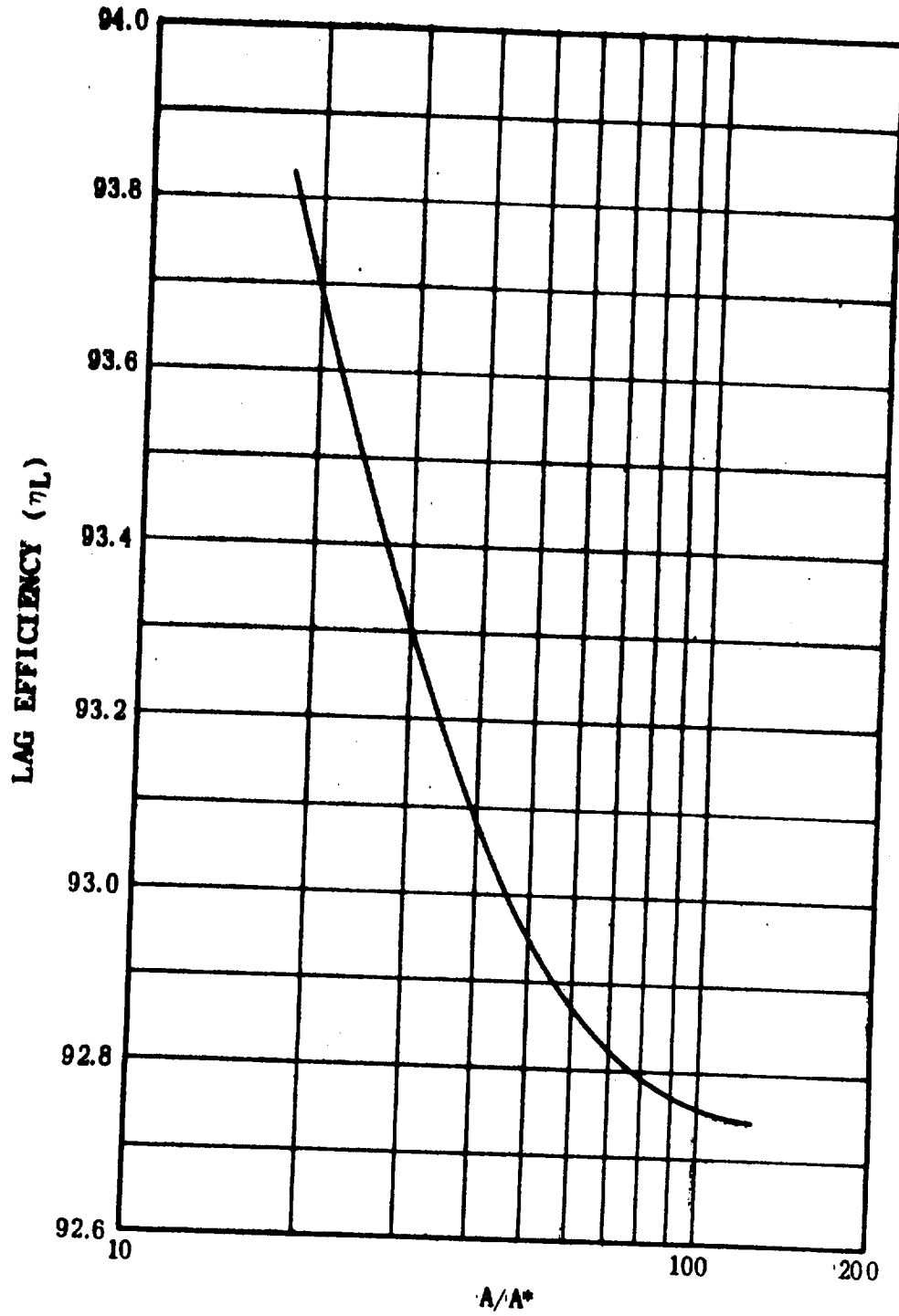


Figure III-8. Rao Nozzle Lag Characteristics.

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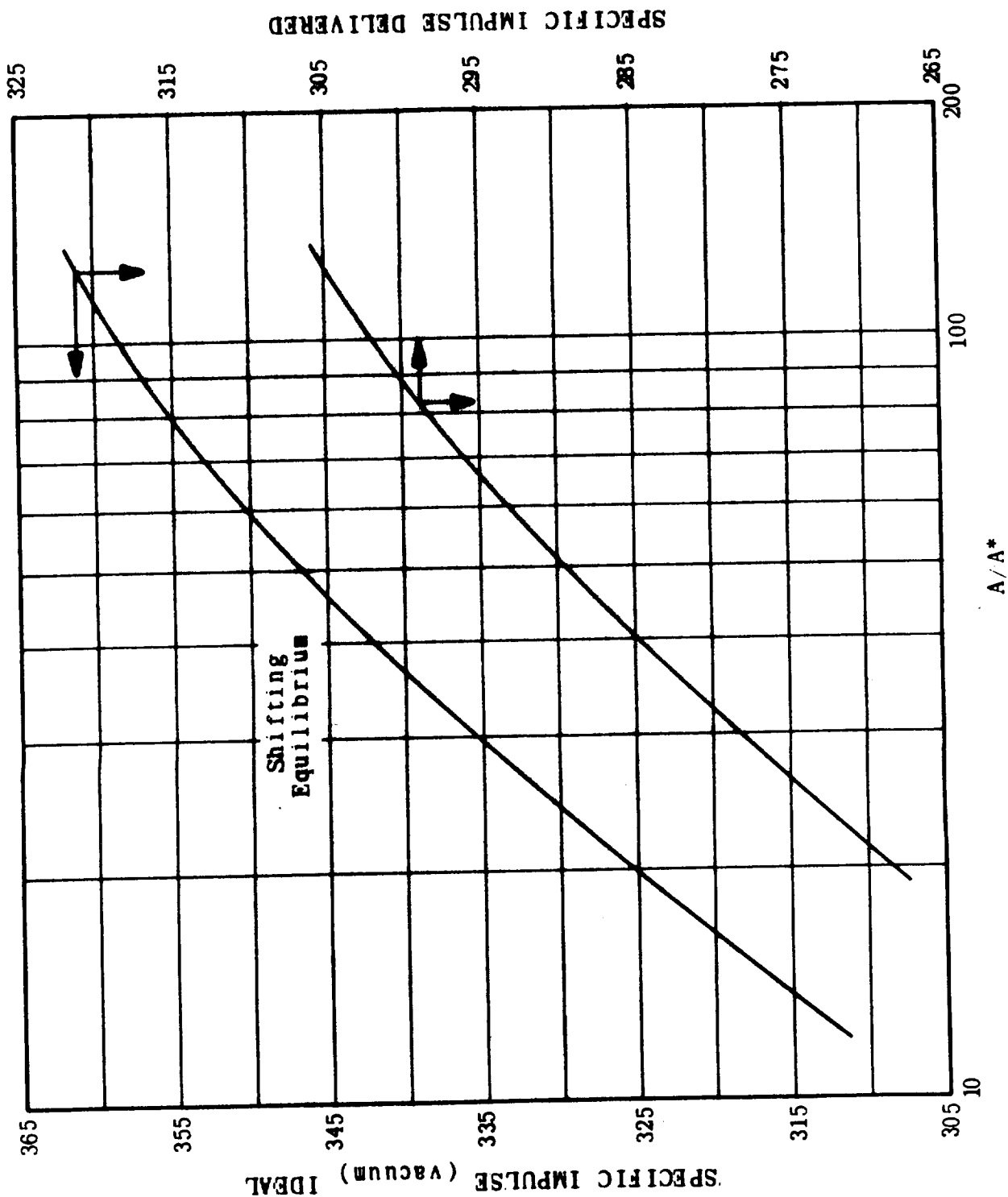


Figure III-9. Motor Performance with Rao Optimum Nozzle.

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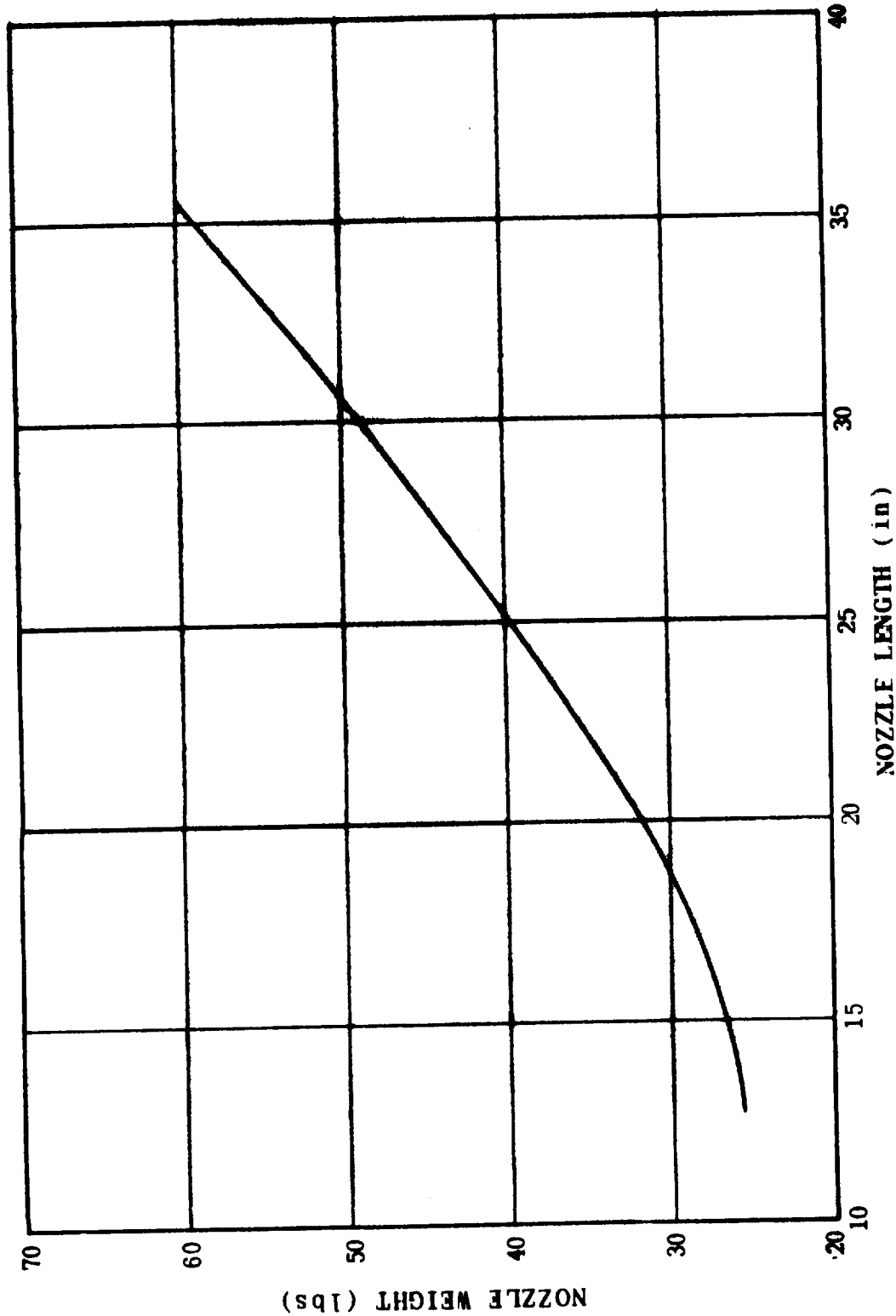


Figure III-10. Structure Weight for Rno Optimum Nozzle.

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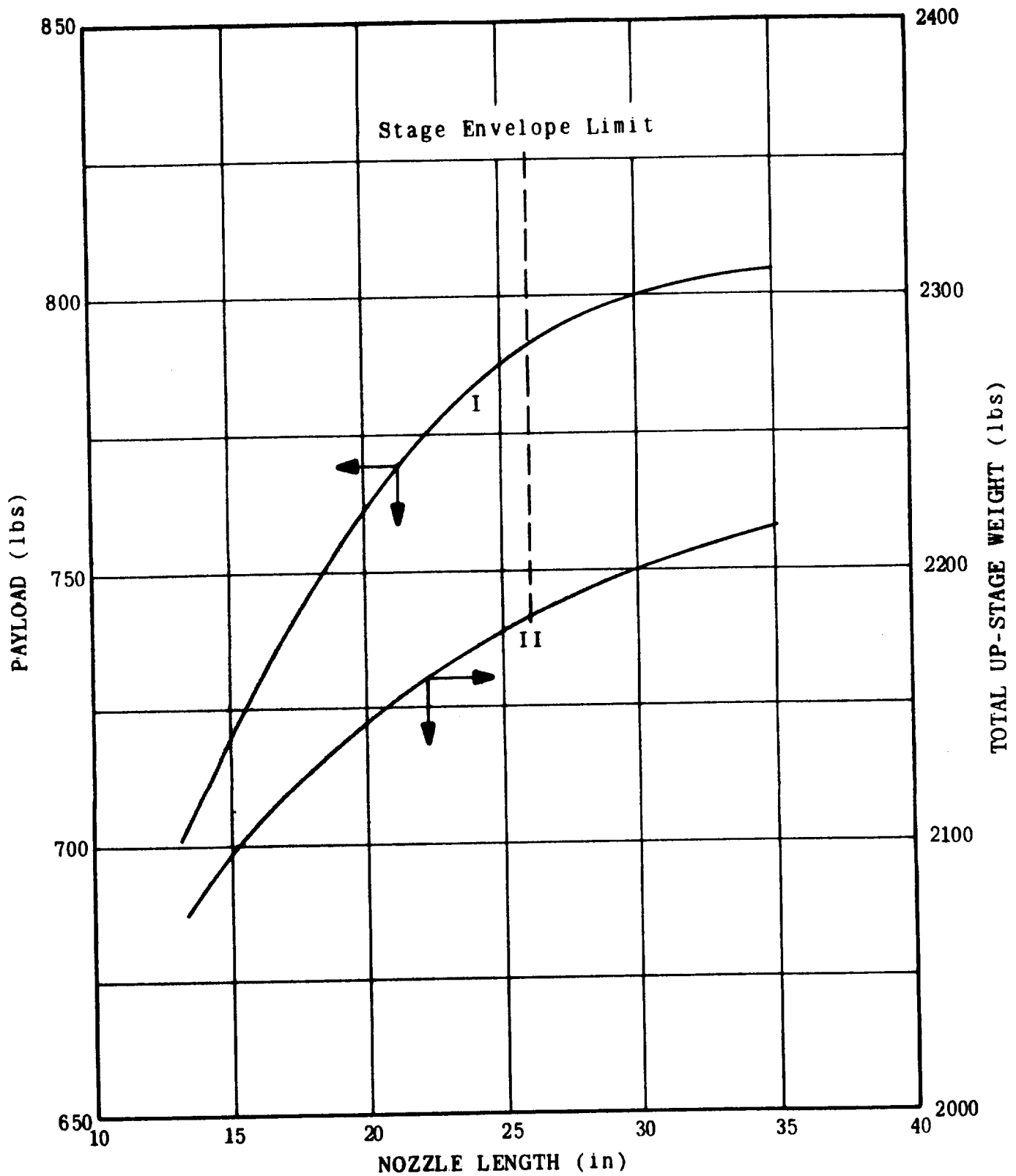


Figure III-11. Up-Stage Weight Variations with Rao Optimum Nozzle.

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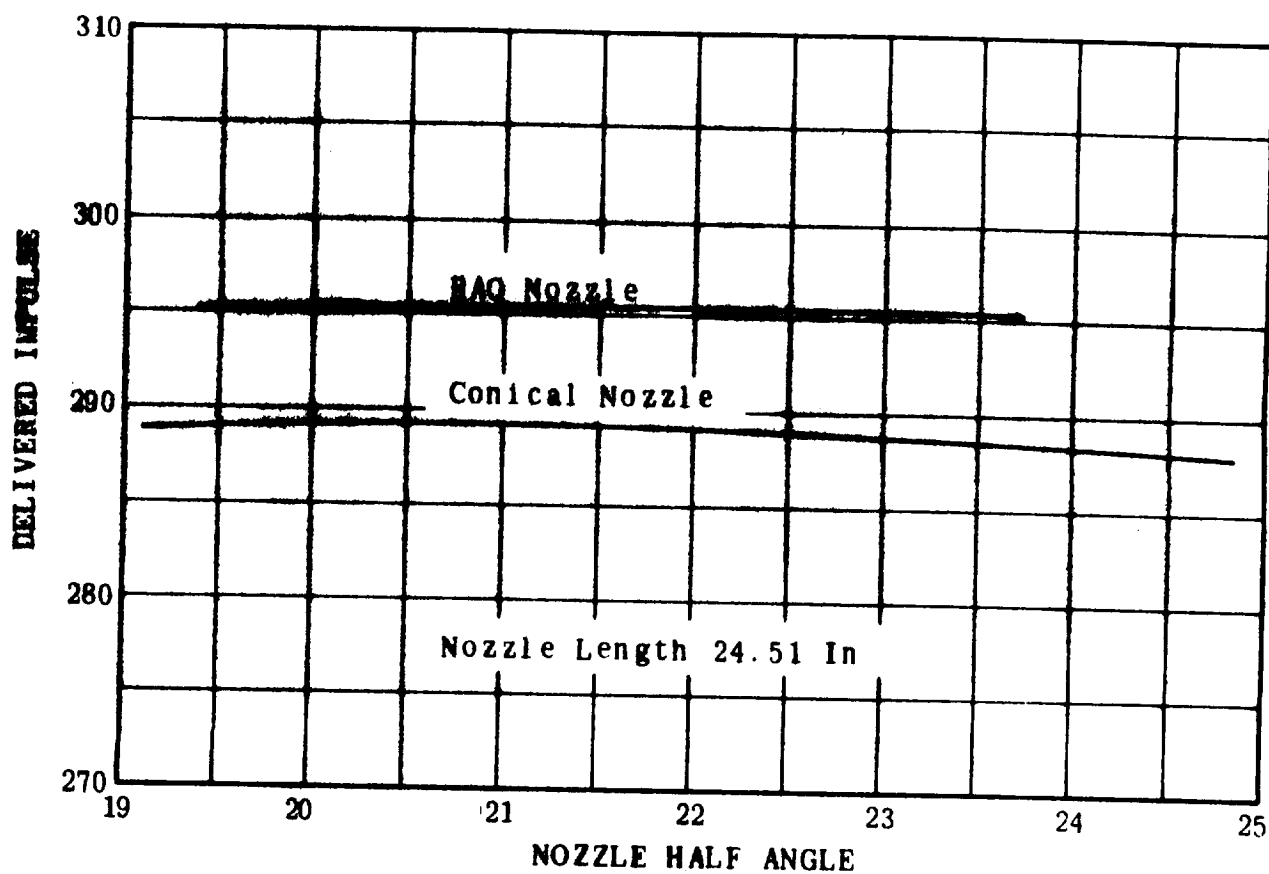


Figure III-12. Comparison of Motor Performance with Rao and Conical Nozzles.

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The lug was fabricated from aluminum sheet, formed into a conical shape having a flared footing as the contact area, as shown in Figure III-13. The footing was cold bonded to an 8.5-inch-radius titanium hemisphere with F-1000 Film Adhesive, manufactured by the Bloomingdale Rubber Company.

A load was applied to the lug to simulate the actual flight loading conditions. The test lug failed under a load of 1,365 pounds; the actual subscale load, based on an estimate of final motor weight, is 1,250 pounds. Strain-gage data indicated a maximum skin stress in the hemisphere of approximately 7,000 psi, a value in excess of the 5,000-psi stress allowable in addition to the maximum skin stress produced by the motor operating pressure. This 7,000-psi stress could be reduced by increasing and judiciously proportioning the footing area. The cold-bonding technique was shown to be acceptable for use in the full-scale attachment lug design. A detailed analysis of this test is included in pages 26 through 38 of MED Report No. SR 207, attached as Appendix D.

IGNITER

The design criteria established by the Jet Propulsion Laboratory included a provision for an igniter which would reliably ignite the spherical motor under vacuum conditions. It was requested that the vacuum capability of the igniter be demonstrated prior to its use in a static firing. To accomplish this task, igniter components with a proven history of vacuum performance were selected for evaluation. Thus, a U. S. Flare 908B squib was selected because of its relatively high no-fire current of 0.7 ampere and because it had been extensively tested and qualified by the U. S. Flare Division of Atlantic Research Corporation and by the Aerojet Corporation for use at high altitudes on the Titan missile. For the main ignition charge, it was decided to use either the standard U. S. Flare 2D boron-potassium nitrate pellets or pellets composed of boron and barium chromate. The former was selected as a candidate material because of its successful history of extensive

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use throughout the igniter industry in a variety of applications. The boron-barium chromate material was chosen because it produces relatively small quantities of gas and would therefore be less sensitive to altitude effects.

The initial igniters were toroidal in shape and constructed from a nylon tube loaded with two squibs and the candidate pellet charges. The nylon tubing was 5/8 inch in outside diameter and 3/8 inch in inside diameter and was bent into a circle approximately 6 inches in diameter. Ten holes were drilled into the tube at such an angle that the emerging flame would impinge on the propellant surface at the extreme after end of the motor behind the submerged nozzle cone. In this manner, the remaining propellant surface would be ignited as the initial gas products flowed around the back of the cone to the exhaust side of the nozzle. The igniter was designed to be bonded to the nozzle of the spherical motor in such a position as not to interfere with the propellant grain.

An initial series of five igniter firings were conducted at sea level to determine the most favorable of the candidate charges. The igniters were all fired vertically; three of the firings were visually observed. A flame pattern approximately 8 feet high emerged almost immediately upon application of current. The physical integrity of the nylon tube was not disturbed. As a result of these tests, a charge of between 10 to 15 grams of 2D pellets was selected. The boron-barium chromate material was rejected because of insufficient reproducibility. Two other igniters were then loaded and successfully fired under vacuum conditions in an altitude test chamber. As the 17-inch spherical motors were static fired, however, it became apparent that the ignition system described above was resulting in excessive ignition delays. Therefore, it was decided to develop an integral nozzle closure and igniter to correct this problem. Because of the lack of funds, however, it was impossible to complete this portion of the igniter development program.

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Figure III-13. Subscale Attachment Lug Tested to Failure at 1365 Pounds.

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CONCLUSIONS

At the termination of developmental testing, all of the major motor components, except the igniter, had been proven acceptable for incorporation into the prototype spherical motor design. The titanium case design was proven by stress analysis and hydrostatic pressure testing and by the success of the heavy-walled subscale motors in static test. The V-44 insulation liner was also shown to be satisfactory in the static firing program. The design of the nozzle for the 36-inch spherical motor was modified so that the retention flange is included as part of the diffuser section rather than as an integral part of the submerged nozzle cone. Hence, the cause of nozzle expulsion problems has been eliminated. It is expected that a satisfactory integral nozzle closure and igniter could be developed with little difficulty.

A propellant mass fraction of 0.922 is currently calculated for the prototype 36-inch spherical motor assembly. A breakdown of component weights is listed below.

<u>Component</u>	<u>Weight (lb)</u>
Motor Case	42.5
Studs	0.4
Nuts	0.1
Nozzle Retention Ring	8.5
Nozzle	39.0
Insulation	17.5
Propellant	1,280.0
Igniter	<u>0.3</u>
	1,388.3

A further weight reduction in nozzle components is considered to be a feasible approach in attaining an even higher fraction.

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IV. STATIC FIRING OF SUBSCALE MOTORS

SUMMARY OF RESULTS

Six heavy-walled steel, 17-inch spherical subscale motors were static fired during the development program. Three of these were loaded with beryllium-containing propellants of the Arcane 40 series; the remaining three contained Arcane 42 propellant, the aluminum analogue of Arcane 40. The latter motors were fired to evaluate motor components and assembly techniques without the introduction of a propellant performance variable. The firing results are summarized below.

<u>Test No.</u>	<u>Firing Date</u>	<u>Arcane No.</u>	<u>Test Results</u>
17S-1	12/27/61	42	Nozzle expelled at 1 second
17S-2	1/17/62	42	Successful; burned for 22 seconds
17SX-1	4/9/62	40	Nozzle insert and liner expelled at 19 seconds
17SX-2	5/31/62	40X	Nozzle expelled on ignition
17S-3	7/10/62	42	Successful; burned for 18.7 seconds
17SX-3	10/11/62	40CX	Case ruptured on ignition after 20-second hang-fire

TEST 17S-1

This first heavy-walled spherical test motor was loaded with Arcane 42, aluminum-containing propellant and fired in December 1961. The

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test objectives were to evaluate the grain design, ignition characteristics, prototype liner material, and general assembly techniques. The motor assembly is shown in Figure IV-1.

Prior to the installation of the thermal insulation liner, the motor case was subjected to a hydrostatic proof-pressure test of 900 psi applied for three minutes. After application of the V-44 asbestos rubber liner, polyurethane foam with a density of approximately 4 lb/ft³ was placed in the after end of the motor. Adjacent to the polyurethane foam on its forward side was bonded a wedge of neoprene foam as shown in Figure IV-1. The exposed surfaces of the motor case liner and the neoprene were then coated with a primer composed of two parts 40X-415 Stanley primer and one part 79R-192 Stanley thinner, both manufactured by the Stanley Chemical Corporation. The first coat was allowed to air dry and then a second coat was applied and cured two hours at 50°C. The same surface was then coated with polyurethane PUX-251 and allowed to cure at room temperature for a minimum of four hours. Arcane 42 propellant batch 137H was cast into the motor and subsequently cured for three days at 125°F plus two days at 140°F.

Static test 17S-1 was conducted on December 27, 1961, at Atlantic Research Corporation's Pine Ridge Test Facility at Gainesville, Virginia. The nozzle was expelled after approximately 1 second of burning. An approximate chamber pressure of 420 psi was attained immediately prior to failure. The cause of failure was attributed at that time to a combination of insufficient engagement of the threads on the nozzle retention studs and of incorrect stud material. The minimum yield strength of the studs was determined to be 45,000 psi rather than the 90,000 psi specified by design. Subsequent testing of nozzles of similar design indicated, however, that the primary source of failure was in the nozzle material itself. It was demonstrated in these tests that the nozzle fails in tension at a chamber pressure of approximately 400 psi with cracks appearing in the asbestos-phenolic immediately opposite the O-ring groove at the after end of the nozzle. The nozzle problem is discussed in detail in the Motor Component section of this report.

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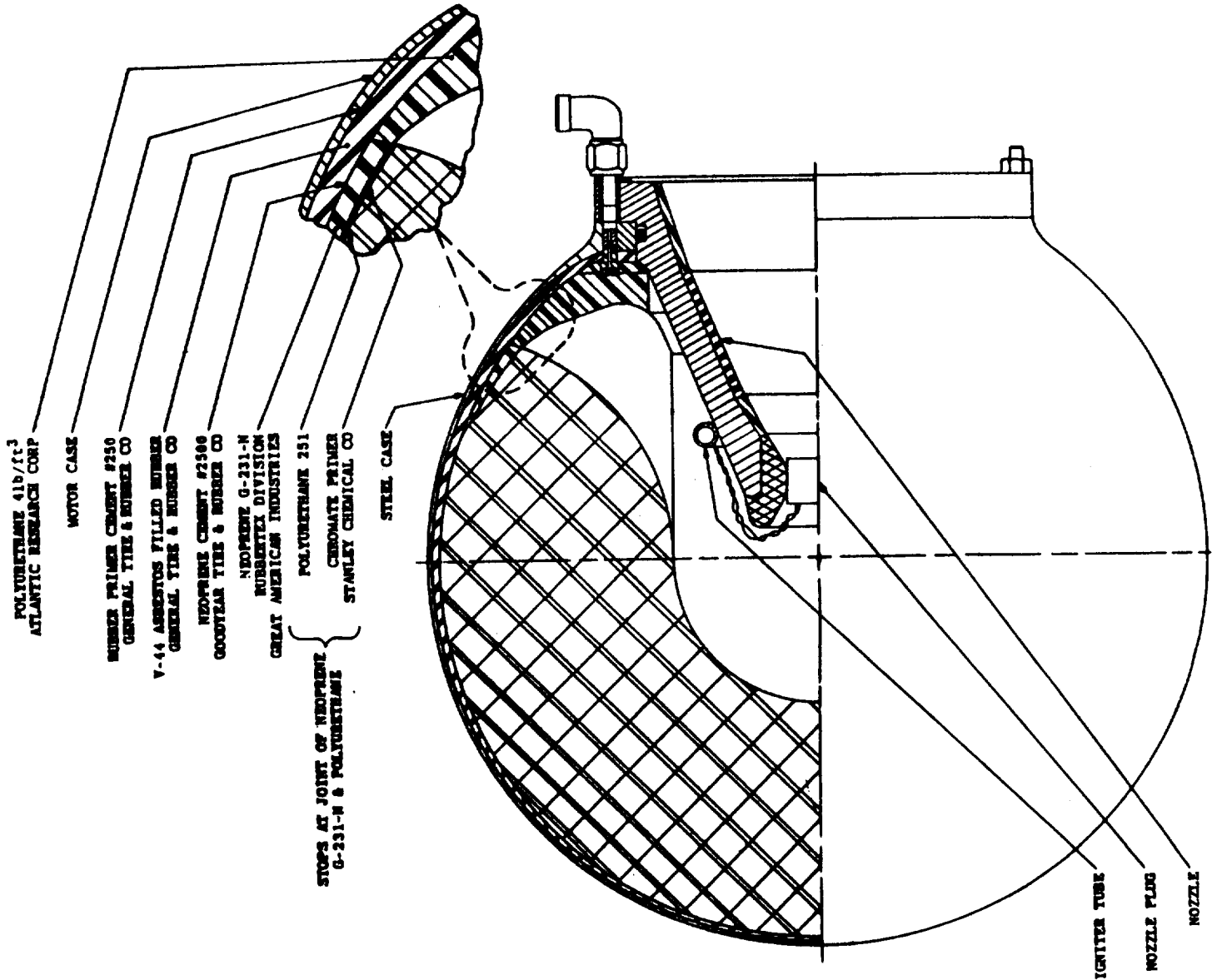


Figure IV-1. Assembly of JPL Heavy-Wall Motor.

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The deficiency in nozzle design was obscured in test 17S-1 since it was impossible to determine whether the nozzle had broken-up prior or subsequent to expulsion. Hence, the only corrective action taken at that time was to ensure that the following motors were fitted with longer studs of design strength.

TEST 17S-2

The second heavy-walled motor, which also contained aluminized Arcane 42 propellant, was fired to evaluate the grain design, ignition characteristics, prototype liner material, and general assembly techniques for the 17-inch spherical motor. The motor assembly of this unit is shown in Figure IV-2.

The case was accepted for test after having passed the 900-psi proof-pressure test. Subsequent to being lined with the V-44 asbestos rubber, the case assembly was inspected and found to be normal and free of defects. The liner in this unit covered the entire internal surface of the case. The thickness varied from 0.060 inch at the forward end to 0.250 inch at the flanged nozzle opening. An insulation ring of 1/4-inch thickness was bonded around the opening to provide an additional heat shield for the nozzle O-ring.

Prior to the casting of the propellant, polyurethane foam, having a density of approximately 4 lb/ft³, was placed in the motor to afford an ignition surface at the nozzle end of the grain. The internal surface of the liner, up to but excluding the foam, was then coated with Stanley primer. The first coat was allowed to air dry, after which a second coat was applied and allowed to cure 2 hours at 50°C. The surface was then coated with two applications of PUX-251 and allowed to cure at room temperature for 4 hours.

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On January 17, 1961, Arcane 42 propellant batch 148H was cast into the motor and subsequently cured for approximately six days at 125°F. No defects in the cured grain were disclosed in visual and radiographic examinations. The motor, after removal of the casting mandrel, is shown in Figure IV-3.

To ensure against a second nozzle failure, the motor was assembled with a heavy steel external nozzle having an ATJ graphite throat insert and an asbestos-phenolic entrance section. The submerged nozzle was not used because of the immediate necessity of evaluating propellant performance and the other motor components in a firing of full duration.

Firing number 17S-2 was conducted on February 7, 1962, at Atlantic Research Corporation's test facility at Corolla, North Carolina. The motor was ignited with the toroidal igniter described in the Motor Component section of this report. After a 3.9-second delay, motor burning initiated and continued without incident for 22.53 seconds. The ballistic data and curves from this firing are presented in Table I and Figure IV-4. The curves exhibited a sharp regressive tendency during the latter half of the firing.

The expended motor case was subsequently sectioned for visual examination; a photograph of the sectioned motor is presented in Figure IV-5. This examination revealed that the web at the base of the internal perforation had burned through prior to the web along the sides of the perforation. This phenomenon explained, at least partially, the regression observed during the latter part of burning. The casting mandrel setup was checked to ensure that the mandrel could not have been displaced during the casting operation. A change in grain design was then effected to increase the web thickness at the base of the perforation by 1/2 inch.

The analysis of expended parts also indicated that a substantial reduction in the thickness of the thermal insulation liner was possible. This change, however, was postponed until more definitive data could be obtained.

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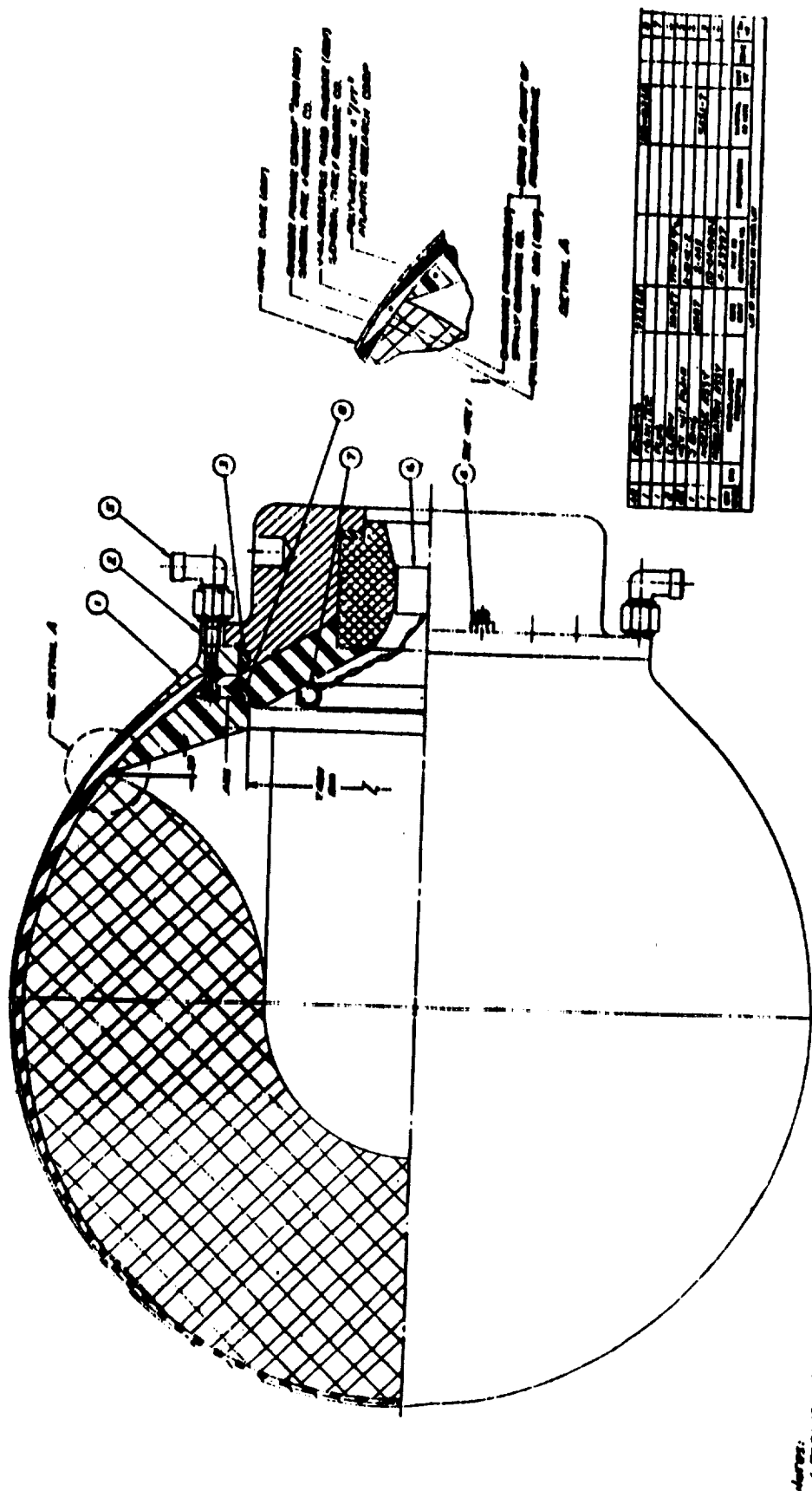
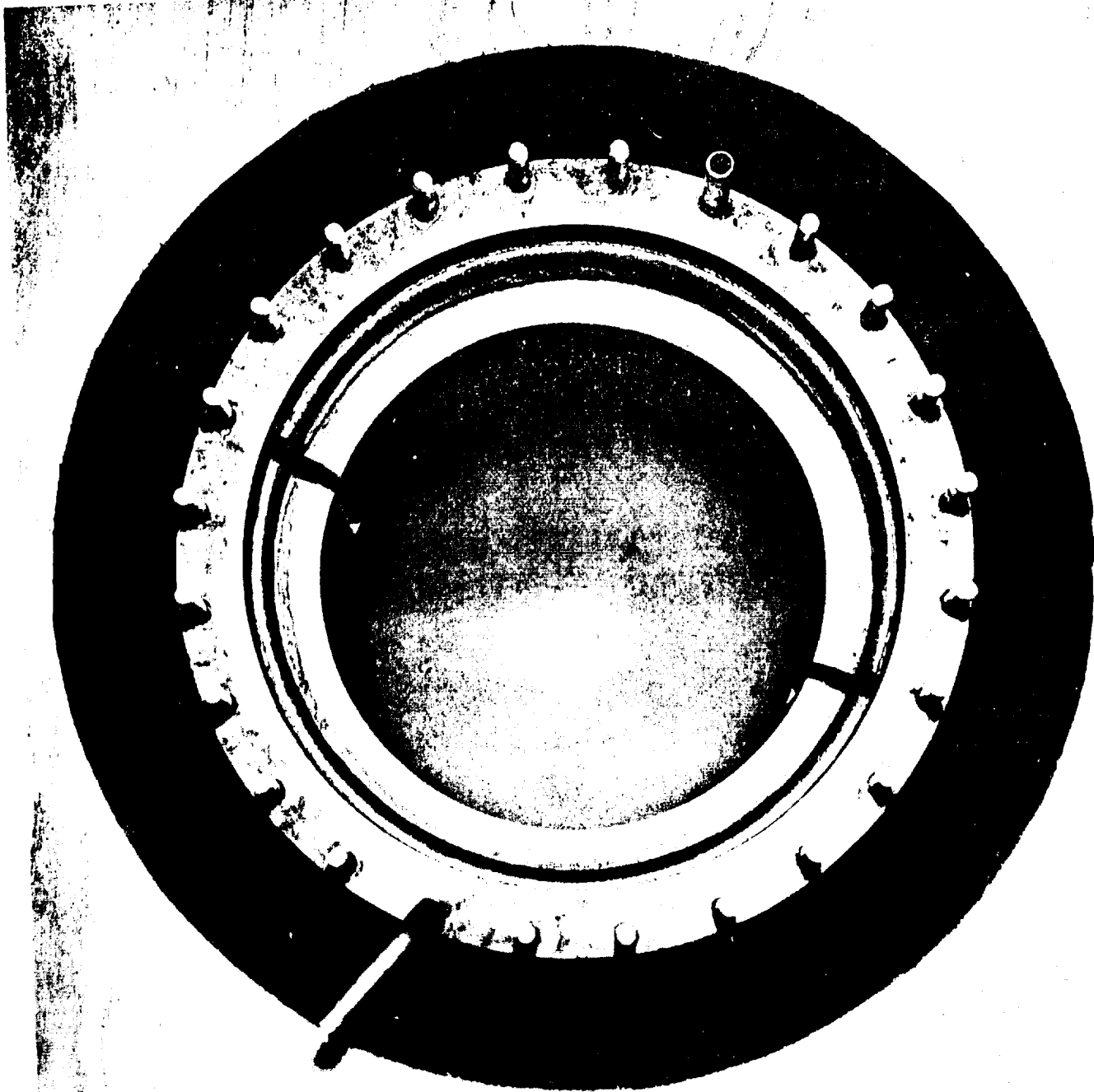


Figure IV-2. Heavy-Wall Test Motor.

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Figure IV-3. Seventeen-Inch Spherical Motor
After Removal From Mandrel.

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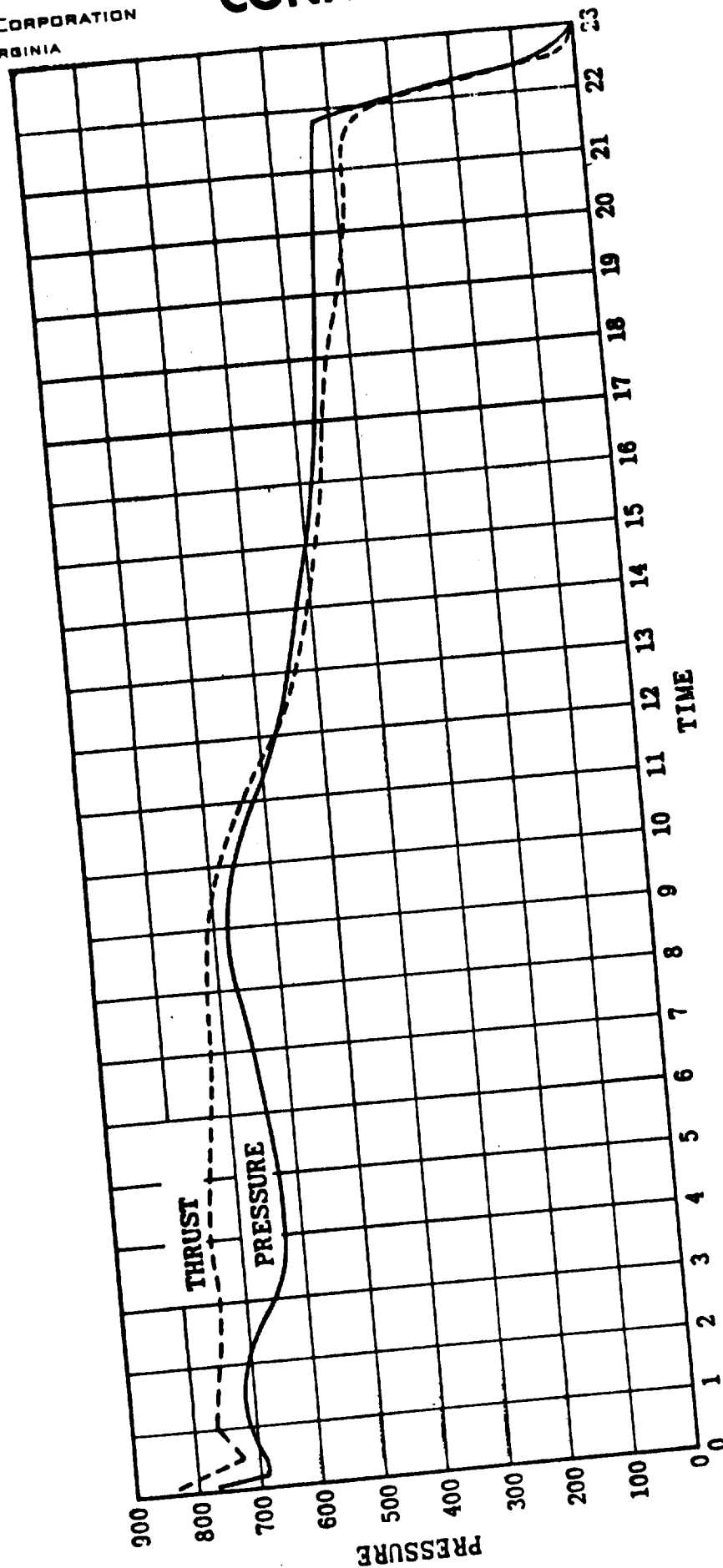


Figure IV-4. Pressure-Time Curve for Static Firing
of 17-Inch Spherical Motor.

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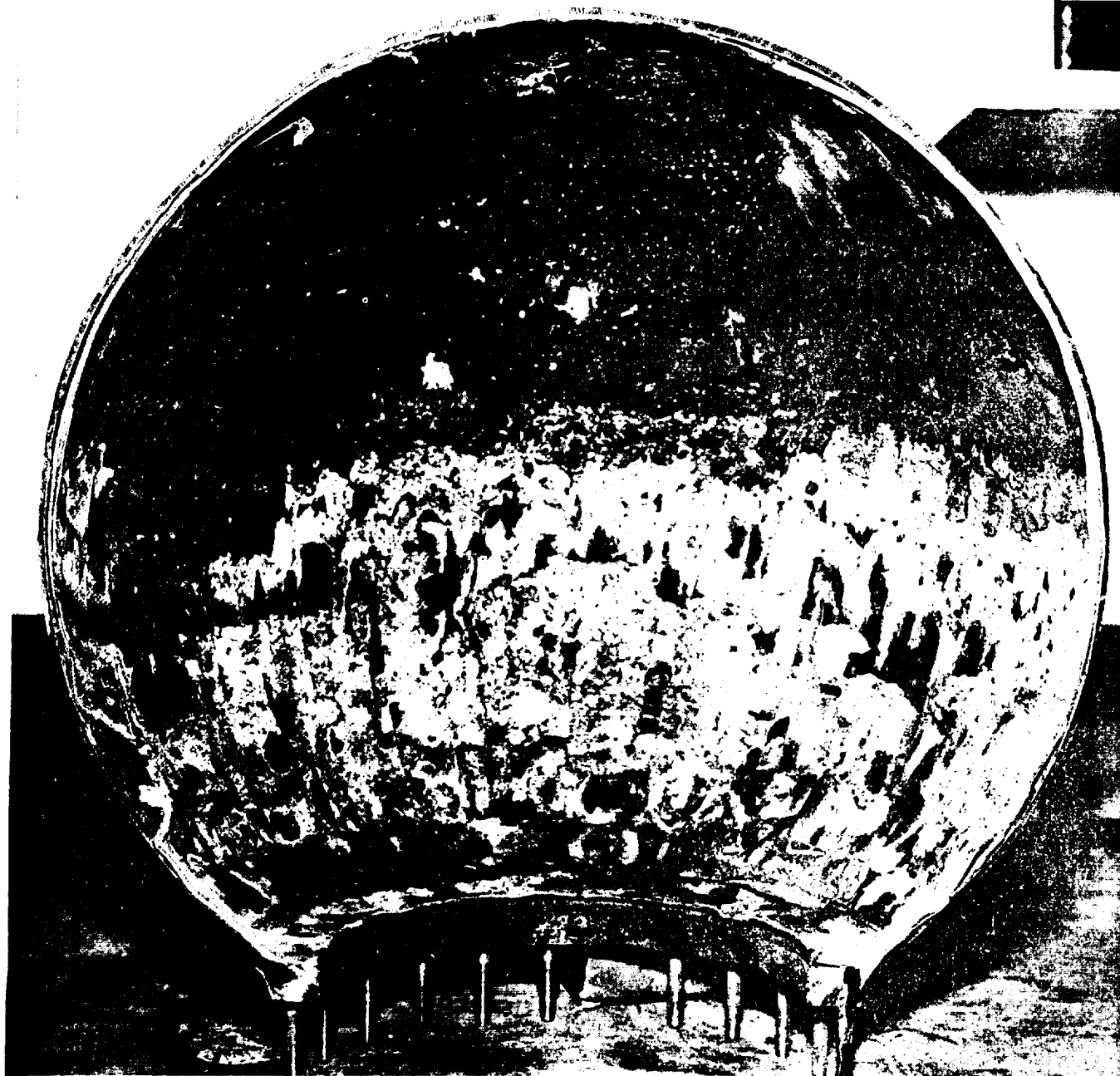


Figure IV-5. Seventeen-Inch Spherical Motor After Firing.

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TABLE I

STATIC FIRING DATA FOR 17-INCH SPHERICAL MOTOR

Grain number	148H-1
Propellant	Arcane 42
Average pressure, psi	576.9
Burning time, seconds	22.53
Burning rate, in/sec	0.227
Discharge coefficient, lb/lb-sec	0.006769
Total impulse, lb-sec	26,988
Average thrust, pounds	1,173
Specific impulse, lb-sec/lb	213
Ignition delay, seconds	3.9

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TEST 17SX-1

The first heavy-walled spherical motor loaded with beryllium-containing propellant was static fired in April 1962. This motor was fired to evaluate the internal ballistics, prototype nozzle material, propellant, liner material, and igniter for the 17-inch-diameter design. The motor assembly is shown in Figure IV-6.

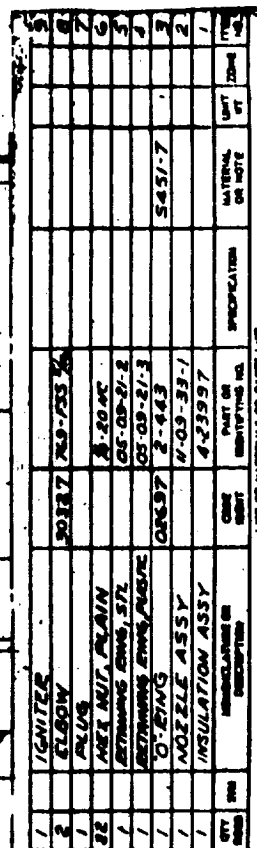
The motor case was found acceptable in visual and dimensional inspections and in the 900-psi hydrostatic pressure test conducted prior to loading. The asbestos rubber liner was installed to the same dimensions as the liner used in the motor of test 17S-2; the lined case appeared to be normal and free from defects. Polyurethane foam, Stanley primer, and PUX-251 were then placed in the motor case in the same manner as in the previous motor.

On March 21, 1962, Arcane 40 propellant batch 205H was cast into the motor and subsequently cured for approximately six days at 125°F. The cure appeared to be marginal, and propellant surface defects were noted as shown in Figure IV-7. These defects were caused by a surface layer of tacky propellant sticking to the mandrel during the curing operation. Although this tacky consistency was eliminated as post curing occurred during exposure to ambient conditions, the propellant grain surface was left in a rather pitted condition.

The nozzle used in this firing was of the same design as that used in firing 17S-1 except for minor differences in the graphite throat insert and liner assembly. The nozzle retention rings, however, differed significantly. The small steel ring used in 17S-1 was replaced with two larger rings: a 1/4-inch-thick phenolic ring supported by a 1/16-inch-thick steel ring. Both rings extended over the asbestos-phenolic flange and the graphite liner; in 17S-1, the ring covered only a portion of the flange.

Static test 17SX-1 was conducted on April 9, 1962, at Atlantic Research Corporation's test facility at Corolla, North Carolina. Ignition was accomplished by means of the prototype torus igniter. After an ignition

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UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES.		APPROVAL BY DATE		ATLANTIC RESEARCH CORP. ALABAMA, VERMONT	
3 YELLOW PAPER		E.R.A. 2-13-42			
4 ANGLES &		V.M.B. 2-13-42			
5 PRACTICE'S		V.M.B. 2-13-42			
2 PLACE DECIMALS &		V.M.B. 2-13-42			
3 PLACE DECIMALS &		V.M.B. 2-13-42			
SURFACE FINISHES ✓		STRESS		MOTOR ASSY. TYPE "C" HEAVY WALL JPL MOTOR	
DO NOT SCALE DIMENSIONS		APPROVED FOR ATLANTIC RESEARCH		COMP. REPORT NO. 93135	
MATERIAL		DRAWN BY		SIZE 8	
		DATE		ED 05-09-17-1	
		SCALE 1/1		1/1	
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Figure 7-6.

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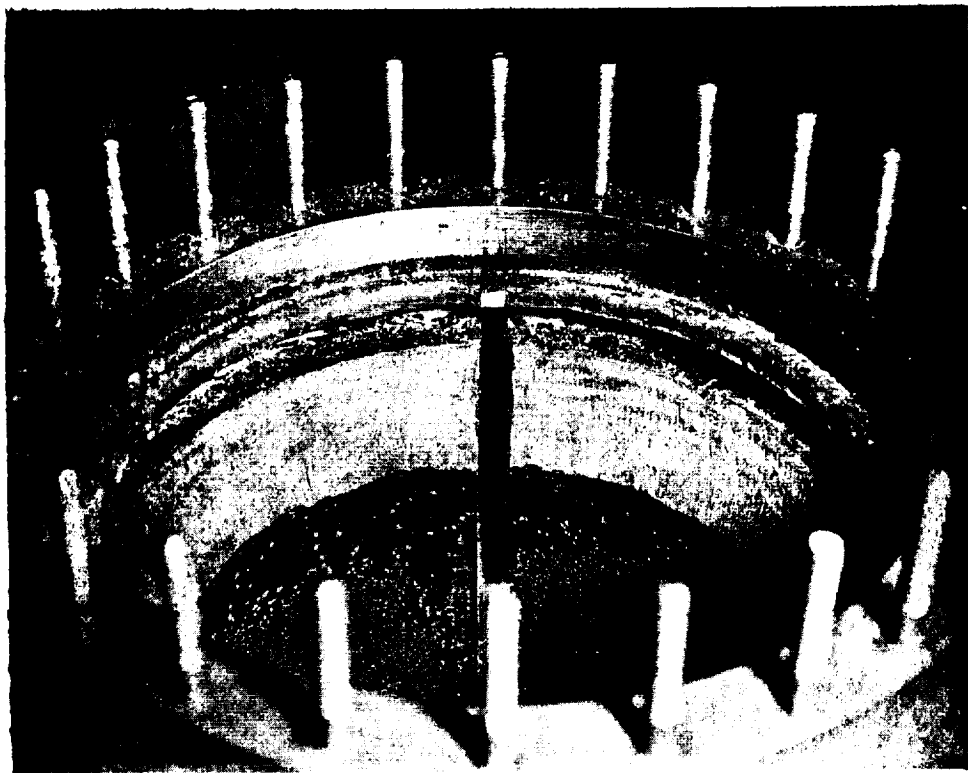
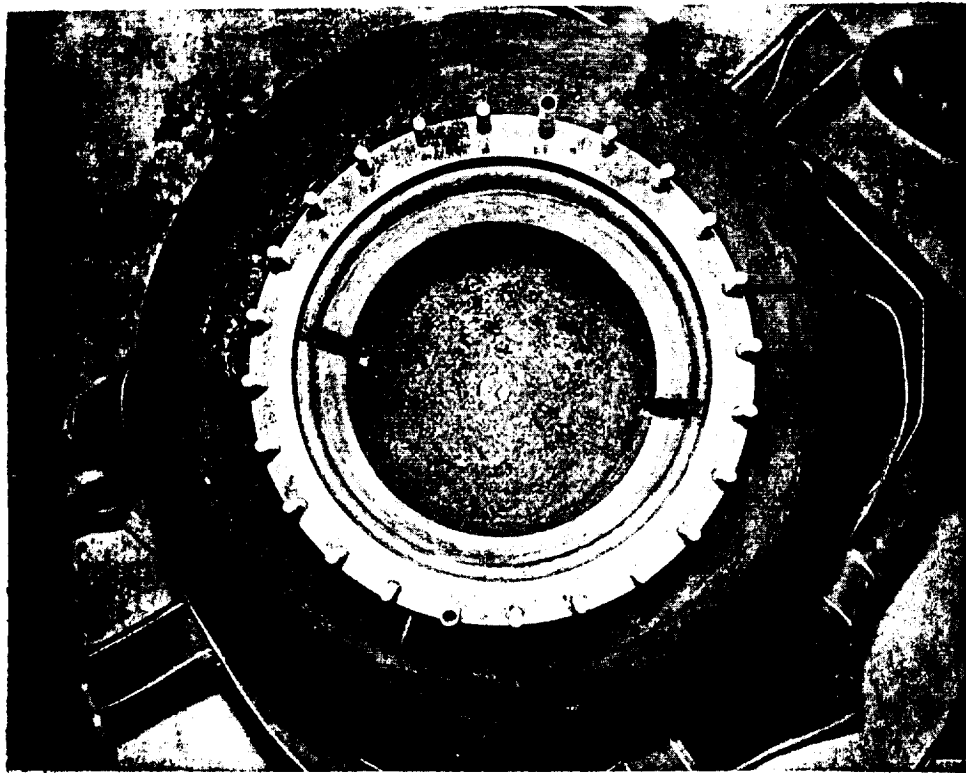


Figure IV-7. Propellant Surface Defects in Motor 17SX-1.

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delay of approximately 1.5 seconds, the motor burned for 19.18 seconds, at which time the nozzle insert and liner were expelled. The thrust-time curve and ballistic data are presented in Figure IV-8 and Table II. No pressure data were recorded for this firing because of a stoppage in the pressure lines. It had been requested by the Jet Propulsion Laboratory that the pressure taps be manifolded so that a nitrogen-purge system could be used immediately after the firing to quench any burning of the insulation. Because of the nature of this system, the pressure lines could not be filled with vacuum grease and consequently became clogged.

A post-firing examination disclosed that the entire entrance portion of the nozzle assembly was lost during the firing, as shown in Figure IV-9. The erratic nature of the firing trace indicates that deterioration of the nozzle and expulsion of material began after approximately 8 seconds of burning. A redesign of the nozzle was initiated to eliminate the graphite liner and replace it with a material with more structural strength. In the first design submitted to JPL for approval, the graphite sleeve was replaced with a graphite cloth bonded to the asbestos-phenolic cone. This design was rejected by JPL because of previous problems encountered in the use of similar nozzles. A second design was then agreed upon by JPL and ARC in which the asbestos-phenolic cone was increased in thickness and the graphite material was completely eliminated. No change was made in the surface configuration of the phenolic cone. This design also incorporated the AHDG graphite throat insert and nozzle entrance section as an integral part to be bonded to the phenolic cone. The interface of the phenolic and graphite is then wrapped with graphite cloth for thermal protection and to prevent dislocation of the insert during firing. The approved nozzle design is shown in Figure IV-10.

A reduction in the thickness of the motor thermal insulation liner was effected after post-firing inspection again disclosed that the insulator was more than adequate. The increase in web thickness at the bottom of the grain perforation appeared to have resulted in uniform burning characteristics.

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TABLE II
BALLISTIC DATA SUMMARY
17-INCH SPHERICAL MOTOR TEST 17SX-1

Arcane 40 Propellant

Grain No. 205H-1

Ignition Delay, t_d , sec	1.458
Ignition Time, t_i , sec	0.172
Burning Time, t_b , sec	19.209
Action Time, t_a , sec	21.699
Time to Failure, sec	19.182
Total Impulse, I_a , lb-sec	23,216
Specific Impulse, I_{sp} , lb-sec/lb	195.85
Average Thrust, F_a , pounds	1,070
Maximum Thrust, F_{max} , pounds	1,240

Definitions

- t_d , time from ignition current applied to first indication of thrust on startup
- t_i , time from first indication of thrust to 75 percent maximum thrust on startup
- t_b , time from 75 percent maximum thrust on startup to corresponding thrust on tailoff
- t_a , time from 10 percent maximum thrust on startup to corresponding thrust on tailoff
- I_a , impulse over action time limits

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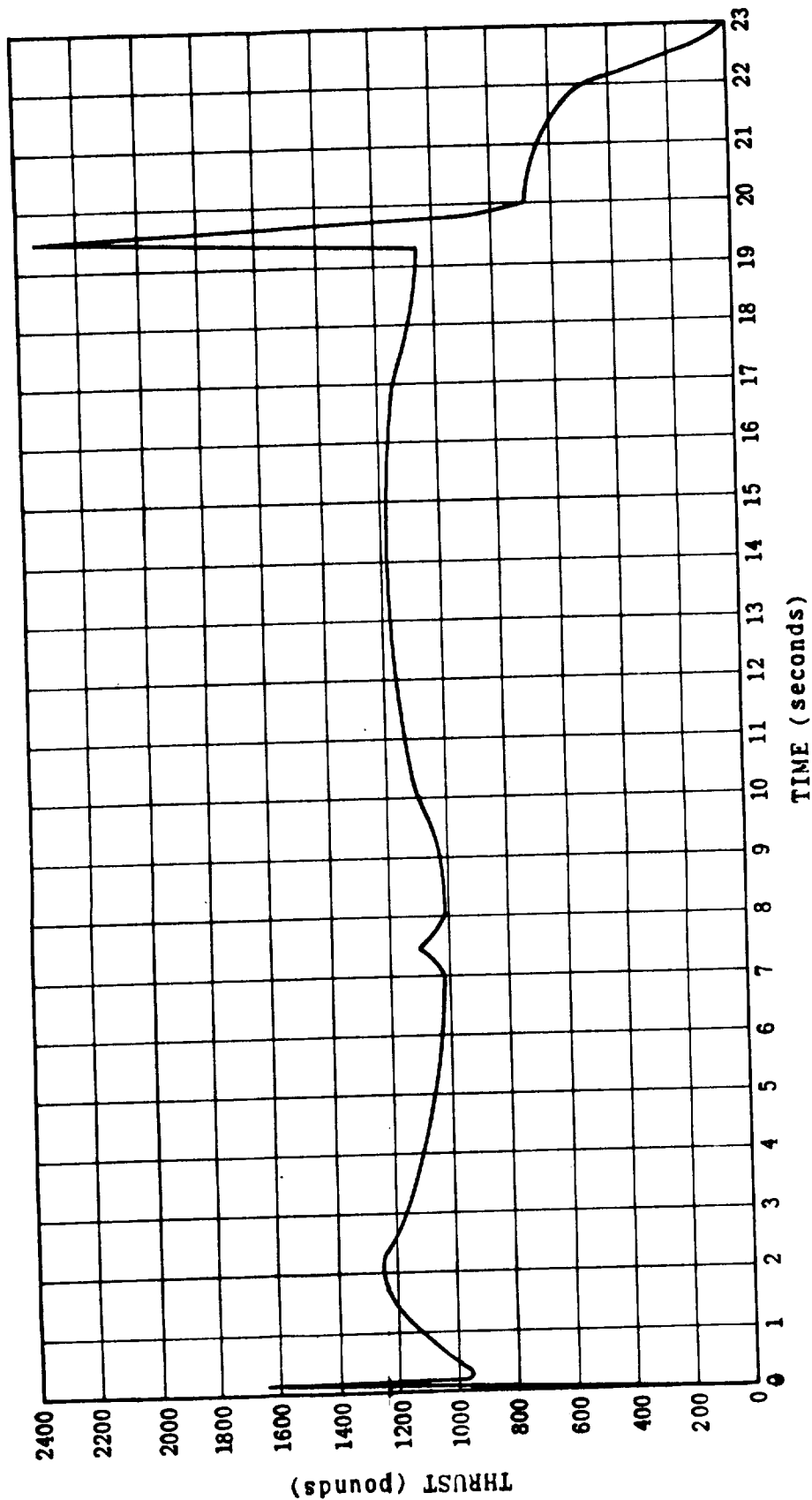


Figure IV-8. Thrust-Time Curve for Static Firing of
17-Inch Spherical Motor, Test No. 17SX-1.

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**Figure IV-9. Nozzle Insulation from
Firing 17SX-1 with
Missing Forward Section.**

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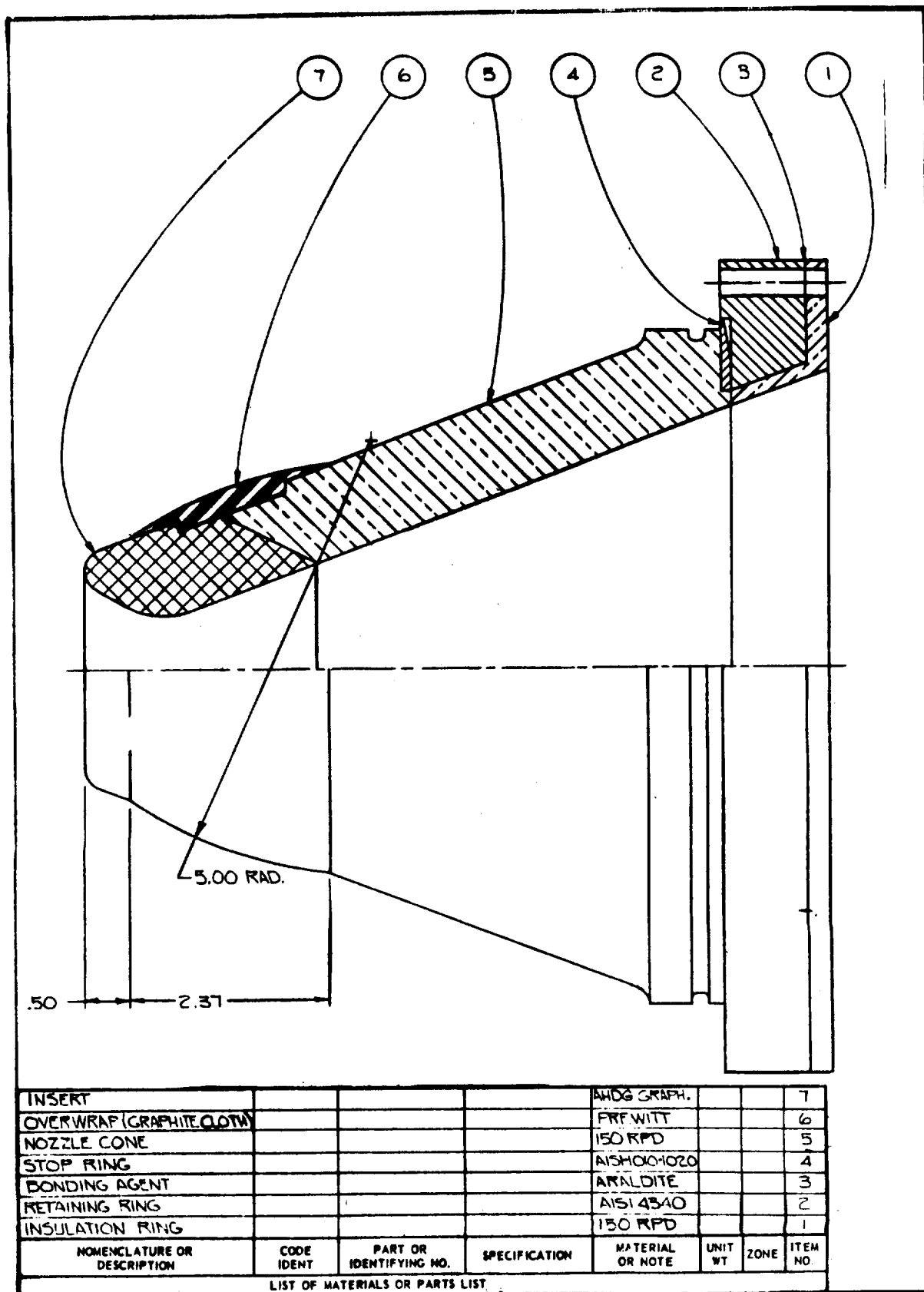


Figure IV-10. Modified Integral-Flange Nozzle.

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TEST 17SX-2

The second heavy-walled spherical motor loaded with beryllium-containing propellant was static fired in May 1962 in test 17SX-2. The assembly of this motor is shown in Figure IV-11.

The motor case was found to be acceptable in visual and dimensional inspections and in a hydrostatic pressure test performed by the manufacturer. The motor case was lined with V-44 asbestos rubber; a visual inspection showed the lined case to be normal and free from defects. In contrast to previous units, the rubber insulation did not cover the entire internal surface of the motor case. The thickness of the insulation varied from 0.125 inch at the motor case flange to approximately 0.010 inch at a location approximately 11.125 inches below the flange. The lower quarter of the surface area of the motor case was left uncovered. In addition, there was a 1/4-inch ring of V-44 insulation material bonded in the flanged opening to serve as a thermal protector for the nozzle O-ring. The polyurethane foam, Stanley primer, and PUX-251 were then installed in the case assembly in the same manner as previously discussed.

On April 24, 1962, beryllium-containing Arcane 40X propellant batch number 231H was cast into the motor and subsequently cured for three days at 125°F and seven days at 135°F. The grain cure appeared to be satisfactory. A radiographic examination of the loaded motor showed no evidence of propellant separation or cracks; however, there were indications of porosity in one area of the grain. The extent of the porous area was not considered sufficient to be detrimental to motor performance. The motor incorporated a torus igniter containing two U. S. Flare 908B squibs and 18 grams of U. S. Flare 2D ignition pellets. The nozzle was of the revised design described under test 17SX-1 and shown in Figure IV-10.

Static test 17SX-2 was conducted at Atlantic Research Corporation's Corolla, North Carolina, facility on May 31, 1962. After approximately a 2.1-second ignition delay, the motor ignited and the heavy nozzle was immediately

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expelled. The propellant subsequently burned for approximately 2 minutes at ambient conditions, and eventually burned a hole through the motor case wall. The nozzle was broken between the conical body and the flange. The conical portion, shown in Figure IV-12, was found approximately 300 feet from the test stand, but the flange portion was not recovered immediately after the test. A small piece of the flange was later recovered and is shown in Figure IV-13.

The available ballistic data and the pressure and thrust versus time curves are shown in Figure IV-14. The data correlation between the two pressure transducers was not satisfactory. One transducer indicated a peak pressure of approximately 750 psia; the other recorded a peak of approximately 1,100 psia. In view of the fast rise time, it may be assumed that the response from one transducer was lagging the other by a small increment.

To clarify the mode of failure in this test, two nozzles of the design used in 17SX-2 were hydrostatically pressure tested as discussed in Section III of this report. These tests indicated that tensile failure of the nozzle occurs at a chamber pressure of approximately 400 psi. Hence, the nozzle was redesigned to incorporate a heavy steel retaining ring in place of the integral flange. A nozzle of this design was fabricated and successfully subjected to a hydrostatic pressure of 900 psi.

TEST 17S-3

The purpose of firing 17S-3 was to evaluate: (1) the redesigned nozzle and retention system; (2) a new integral nozzle-closure and ignition system. The heavy-walled steel, 17-inch spherical motor was loaded with Arcane 42 propellant, the aluminum-containing analogue of Arcane 40X, and fired in July 1962. The motor assembly is shown in Figure IV-15.

To reduce the excessive ignition delays which had been experienced in previous firings, an integral nozzle closure and igniter was developed

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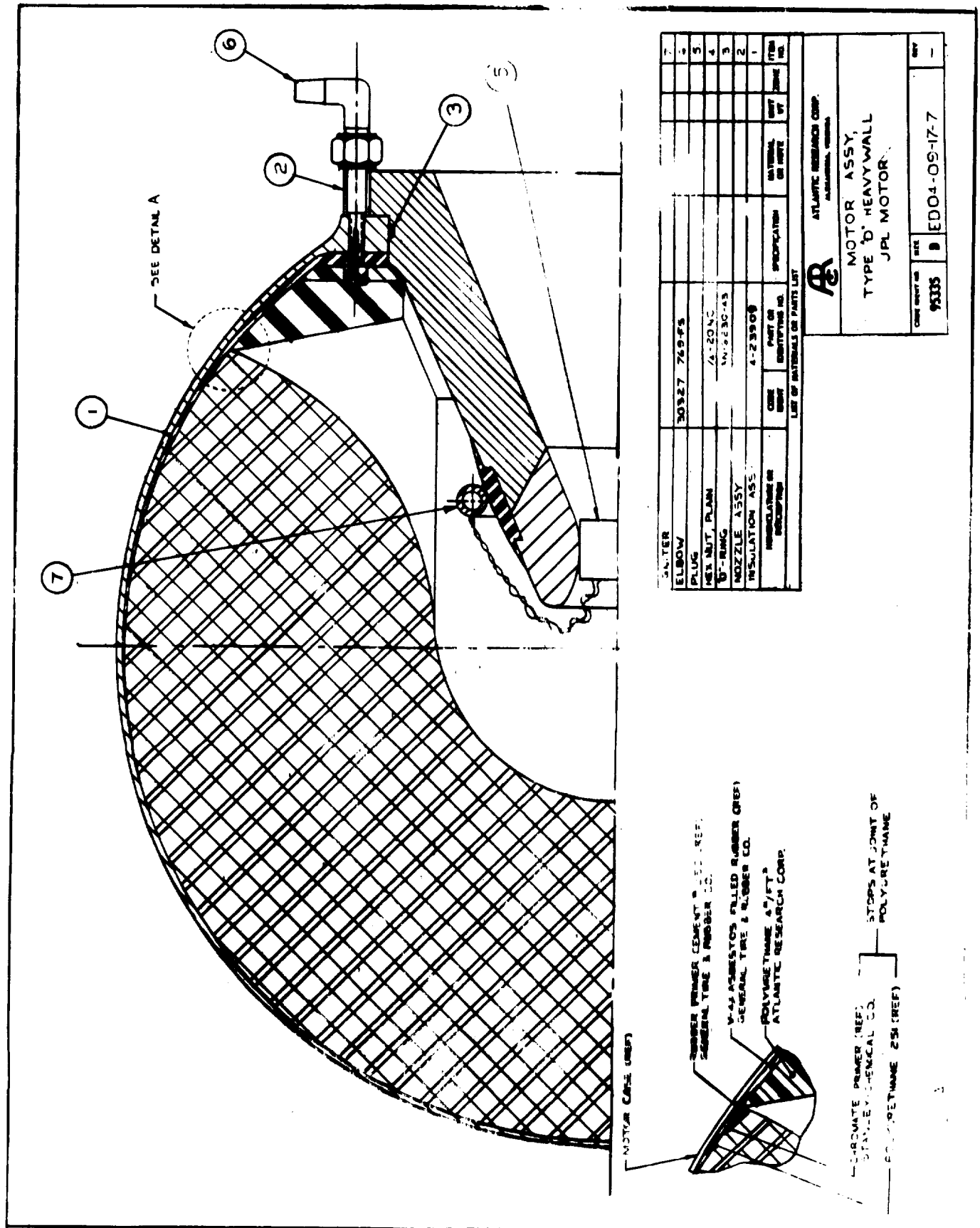
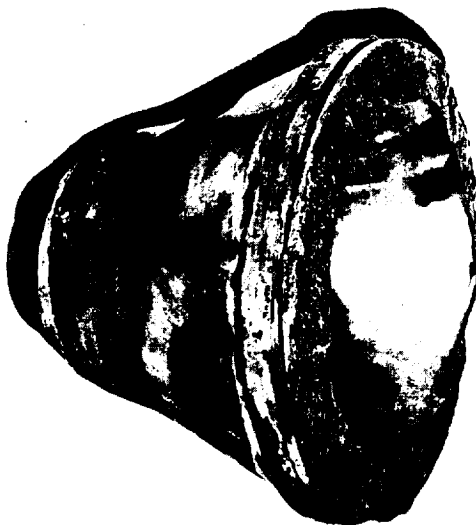


Figure IV-11.

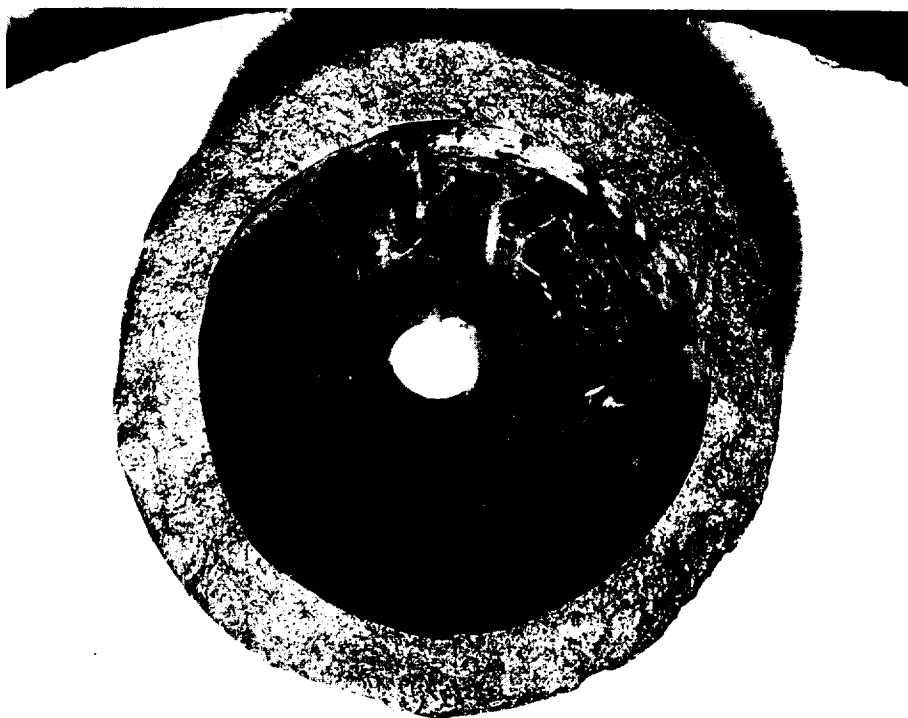
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Figure IV-12. Nozzle from Firing Test 17SX-2 Showing
Break Immediately Above "O"-Ring Groove.

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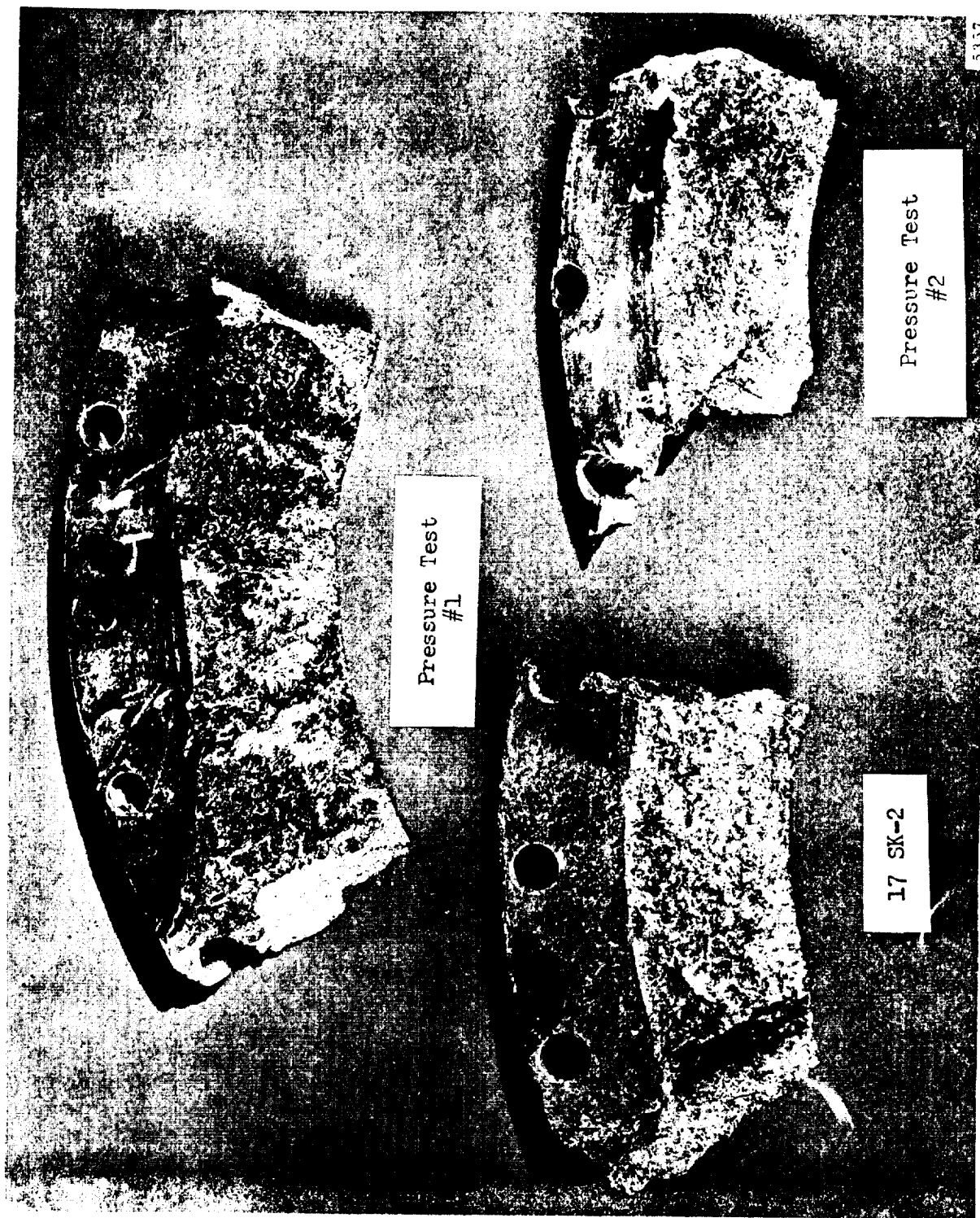


Figure IV-13. Comparison of Nozzle Flange Fragments from Hydrostatic Pressure Test Samples with Corresponding Fragment From Firing 17SX-2.

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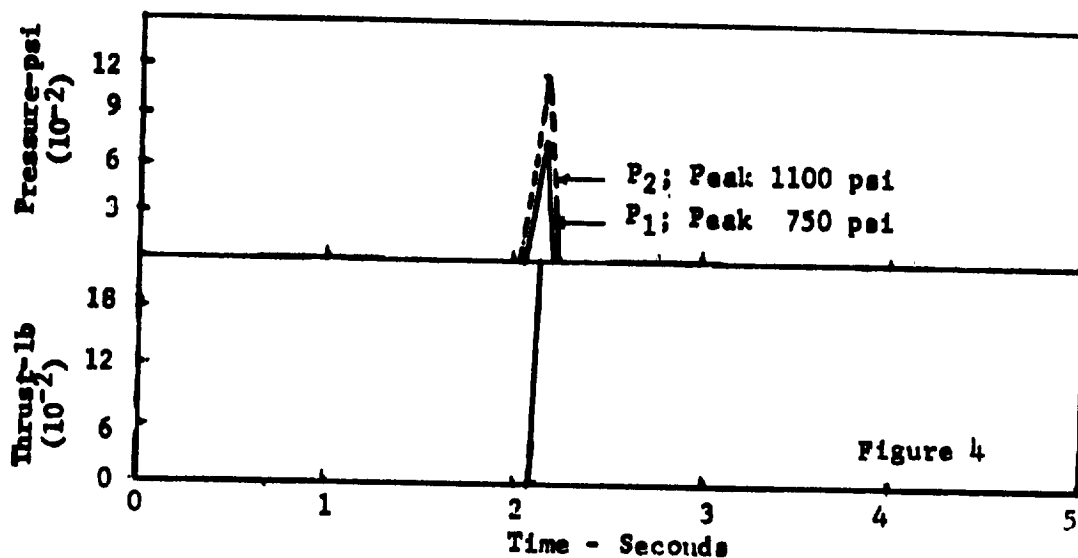
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Arcane 40X Propellant

Grain No. 231-H

Ignition Delay	2.1 seconds
Time to Failure	2.1 seconds
Burning Time after Failure	Approximately 2 minutes
Maximum Pressure	Approximately 1100 psi



Motor Weights:

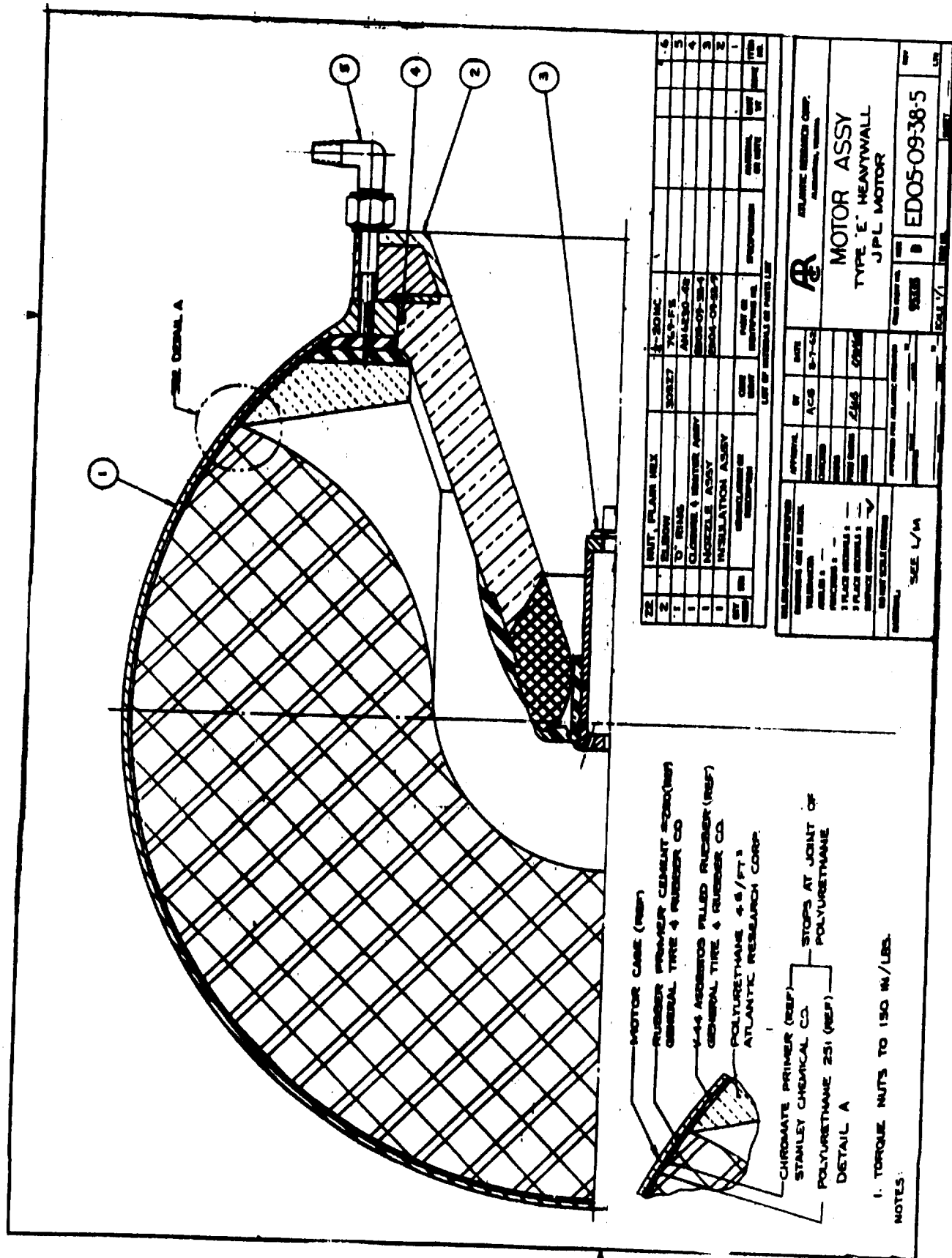
<u>Component</u>	<u>Weight (lbs)</u>
Motor Case Number 15	28.85
Insulation	2.90
Foam and Primer	0.78
Polyurethane (PUX)	0.46
Propellant	<u>122.80</u>
Total	155.79

Figure IV-14. Ballistic Data Summary—17-inch Spherical Motor Test 17SX-2.

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for use on this motor. A system of this kind was designed, fabricated, and successfully tested for functioning characteristics. The assembly is shown in Figure IV-16.

The motor case for firing 17S-3 was found acceptable in visual and dimensional inspections and in the 900-psi hydrostatic pressure test. The Jet Propulsion Laboratory requested that the insulation liner used for this motor be of the same dimensions used initially in the program to reduce the possibility of failure by burn-through. The V-44 insulation was installed to the same dimensions as those used for the liner on firing 17S-2, inspected, and found to be acceptable. The polyurethane foam, Stanley primer, and PUX-251 were then installed in the same manner as previously described.

On July 10, 1962, the motor was loaded with Atlantic Research Corporation's Arcane 42 aluminum-containing propellant, batch number 303H. The grain was cured for five days at 135°F to 140°F and subsequently subjected to a radiographic inspection, which revealed no grain defects. Firing 17S-3 was conducted at Atlantic Research Corporation's facility at Corolla, North Carolina, on July 29, 1962, under simulated altitude conditions. The pressure and thrust characteristics, neutral during the first 9 seconds of burning, became predominantly regressive during the latter half of the firing. The pressure reached a peak of 840 psi at 6 seconds and then dropped to 540 psi at tail-off; the average pressure over the action time was 712 psi. The 18.7-second action time was approximately 3 seconds shorter than had been anticipated. This was attributed to the high burning rate resulting from the unexpectedly high pressure experienced during the first half of the firing and because the physical dimensions of the grain were smaller as a result of thicker insulation. The severe regressivity was at least partially a result of nozzle throat erosion. The throat diameter after firing varied from 0.060 to 0.080 inch greater than the initial diameter of 1.304 inches. Ballistic data are presented in Figure IV-17.

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The nozzle and nozzle retention system maintained their structural integrity throughout the firing. Photographs of the nozzle and of the segmented nozzle body are shown in Figures IV-18 and IV-19. As shown by the latter photograph, the material thickness of the nozzle could be reduced appreciably without affecting motor safety. The char rate of the 150 RPD asbestos phenolic was approximately 0.054 in/sec on the side of the nozzle subjected to exhaust and approximately 0.96 in/sec on the opposite side.

The integral nozzle closure and igniter assembly failed to function as expected: the igniter was expelled too quickly to effect propellant ignition. It was necessary to ignite the motor with approximately 20 grams of U. S. Flare 2D ignition pellets, retained in a polyethylene bag, and a U. S. Flare 908B squib. Had funds been available to continue the development of the igniter, the next step would have been to increase that portion of the igniter which extends past the closure into the motor. Radial holes could then be drilled in the igniter, thereby reducing the load on the closure as the igniter functions.

TEST 17SX-3

The sixth heavy-walled steel, 17-inch spherical motor was fired in test 17SX-3 as a final evaluation of the beryllium-containing propellant system. The motor assembly was identical to that shown in Figure IV-15 for test 17S-3.

The motor case for firing 17SX-3 was found to be acceptable in visual and dimensional inspections and in the 900-psi hydrostatic pressure test conducted prior to loading. The interior of the motor case was subsequently insulated with V-44 asbestos rubber and inspected for any abnormalities or defects. The inspection showed the liner to be normal and free from defects. Again the thick-walled insulation liner was used since it was desirable to keep the variables at a minimum. After the liner was installed, the polyurethane foam, Stanley primer, and PUX-251 were placed

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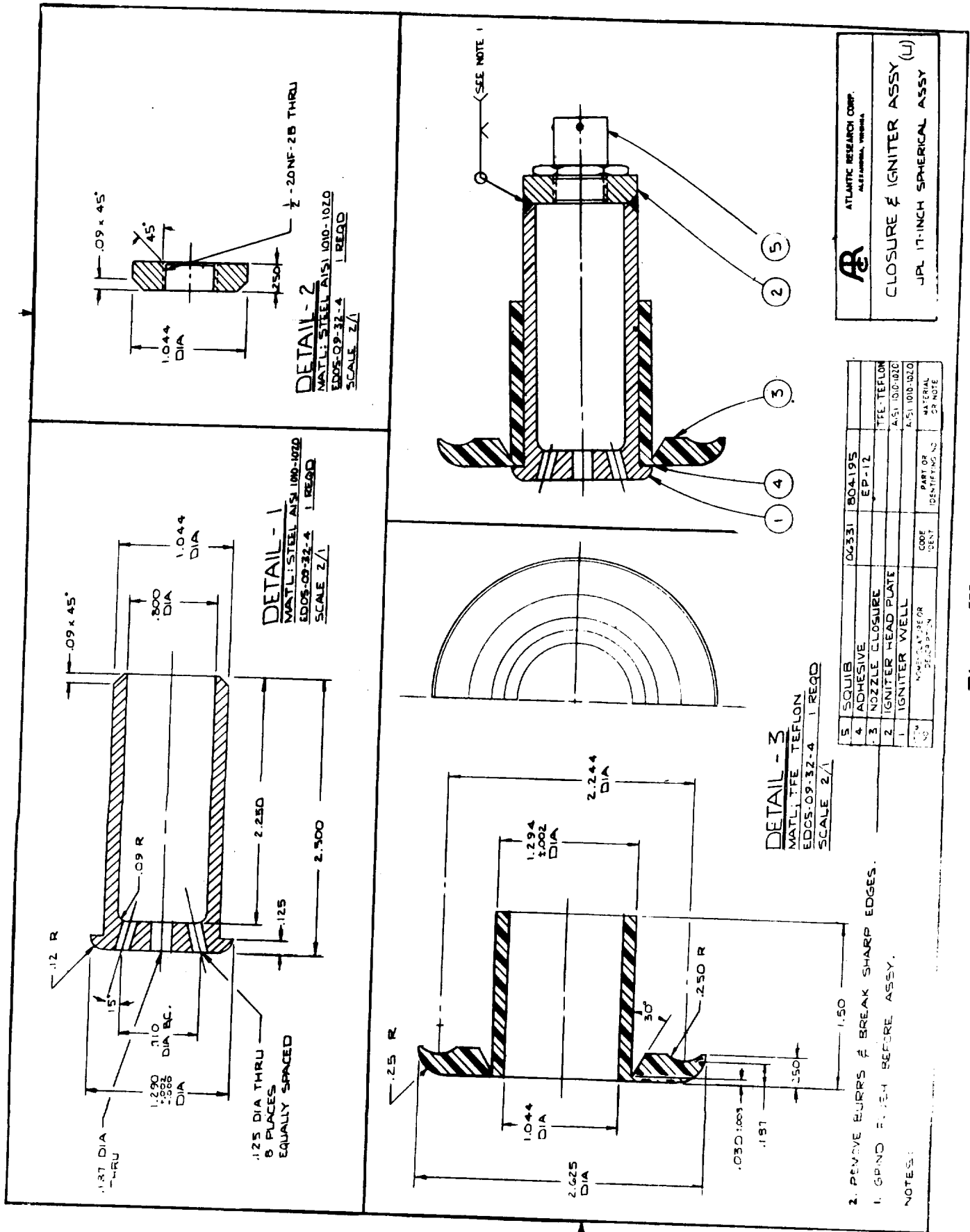
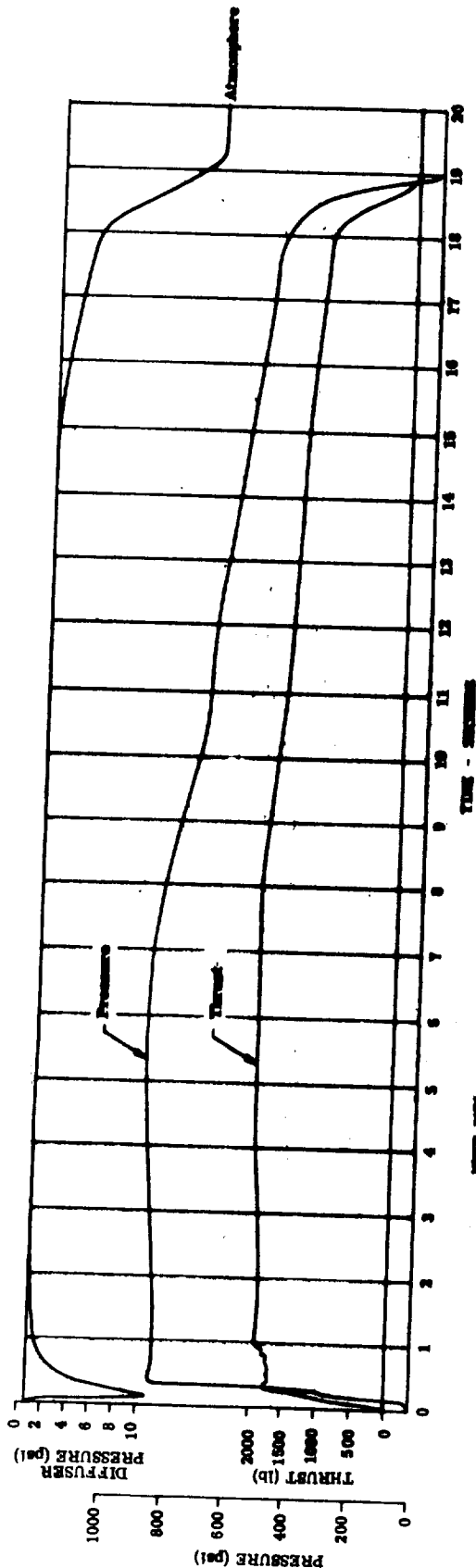


Figure IV-15

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Motor Case Description JPL 17-inch Subminiature Motor-00-20-0

Treatment prior to firing None

Propellant Type Acrylonitrile

Motor Data

Motor	20.00 lb	Motor Reference No.	00000-00-20-0
Insulation	7.00 lb	Motor's Thrust Dia (in)	1.200
Form & P.O.X.	1.00 lb	Motor's Thrust Dia (in)	1.200
Core	1.00 lb	Motor's Thrust Dia (in)	1.200
Core (2)	1.00 lb	Motor's Thrust Dia (in)	1.200
Insulator	1.00 lb	Motor's Thrust Dia (in)	1.200
Propellant	124.00 lb	Motor's Thrust Area (in ²)	0.0001
Density	0.0001 lb/in ³	Motor's Thrust Area (in ²)	0.0001
Motor (AF)	34.3 lb	Motor's Thrust Area (in ²)	0.0001
Nozzle (AF)	7.00 lb	Motor's Thrust Area (in ²)	0.0001
Nozzle No. 10.10 lb		Motor's Thrust Area (in ²)	0.0001
Igniter Description	20 gm 20 Puffs	Motor's Thrust Area (in ²)	0.0001
Igniter Material	alum	Motor's Thrust Area (in ²)	0.0001
Ignition Voltage	24 volts	Motor's Thrust Area (in ²)	0.0001
Ignition Current	amp	Motor's Thrust Area (in ²)	0.0001

Description of motor after firing Normal, no insulation burn through

Ballistic Data

Ignition Delay	sec	0.000
Ignition Time	sec	0.000
Burning Time	sec	0.000
Action Time	sec	0.000
Pressure, Bar	psi	0.000
Pressure, Bar	psi	0.000
Thrust, Bar	psi	0.000
Thrust, Bar	psi	0.000
Total Impulse (N)	lb-sec	0.000
Total Impulse (N)	lb-sec	0.000
Specific Impulse (N)	lb-sec/lb	0.000
Specific Impulse (N)	lb-sec/lb	0.000
Coefficient of Mass Discharge	0.0000	
Specific Impulse Efficiency	0.0000	
Average Effluent Pressure	1.315 psi	
O-O Impulse	31,702 lb-sec	
C _d	4000 lb/sec	

General

Order No. JPL 000007

Contractor: Atlantic Research Corporation

Test No. 00000

Date 20 July 1962

Test Facility Atlantic Research Corporation

Prepared by 20000000

Date 7/5/62

Approved by L. B. Brown

Date 7/5/62

Figure IV-17 Ballistic Data for Firing Test No. 17S-3.

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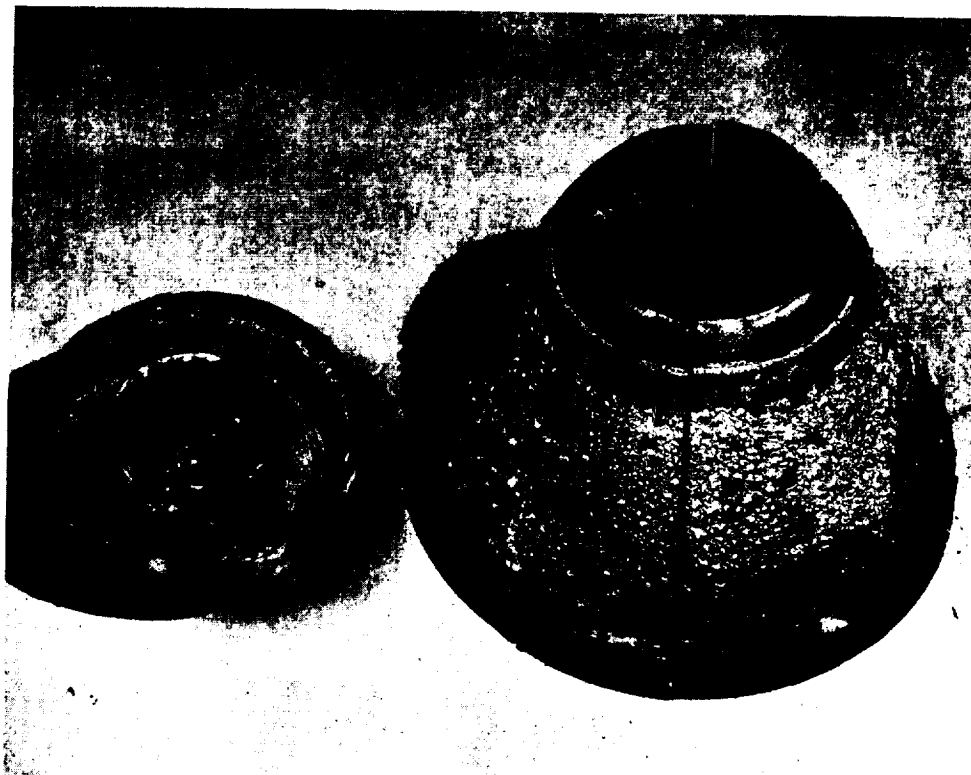


Figure IV-18. Two Views of Nozzle and Nozzle Insert
from Spherical Motor Firing 17S-3.

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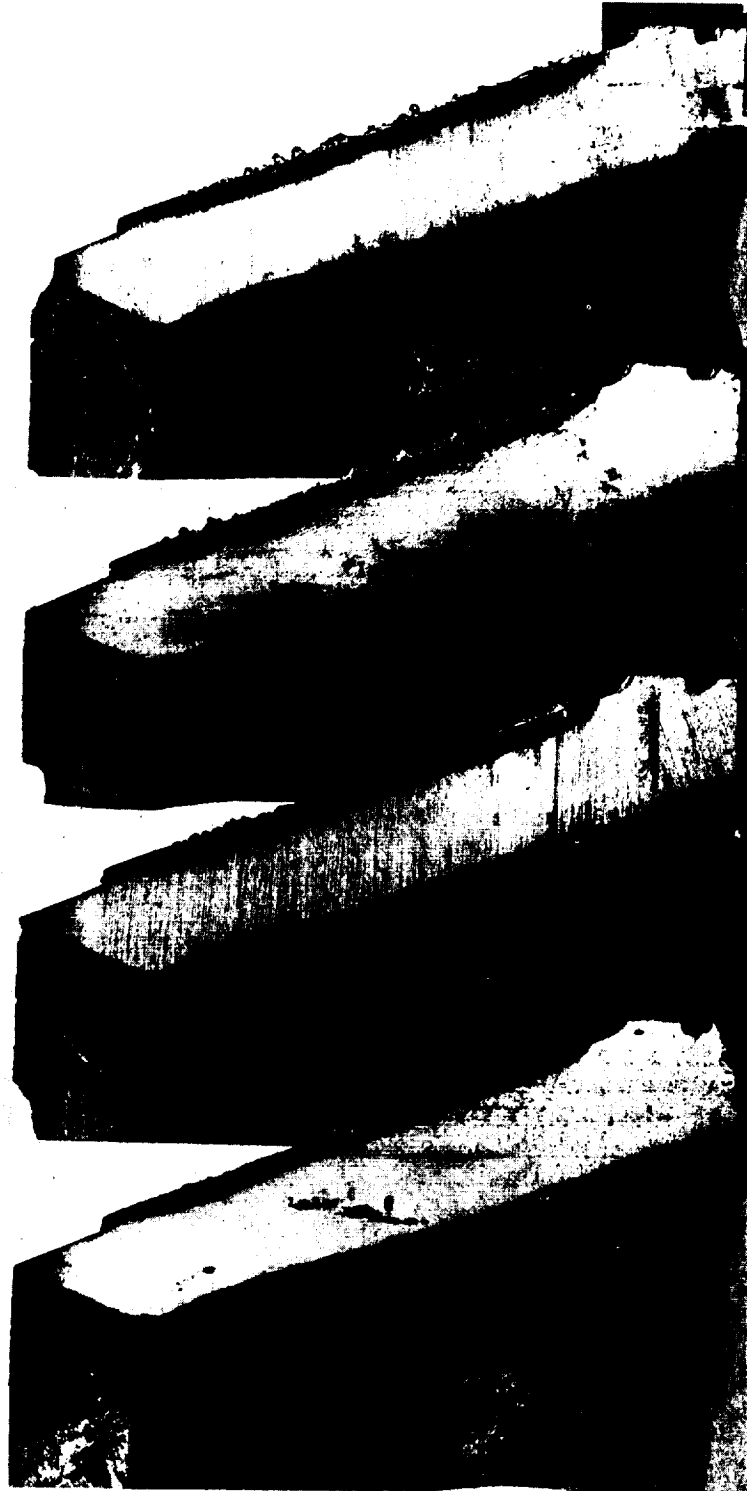


Figure IV-19. Segmented Nozzle from Spherical Motor
Firing 17S-3 Showing Char Thickness.

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into the lined case as in previous motors. The motor was then loaded with Arcane 40CX propellant batch number 349H on August 8, 1962. The grain was cured for five days at 135°F to 140°F. The motor was then subjected to a radiographic inspection, and no apparent grain defects were observed. A nozzle of the same design as that used in test 17S-3 firing was used in this motor, even though a material reduction could have been safely accomplished. The igniter consisted of 20 grams of U. S. Flare 2D pellets, retained in a polyethylene bag, and two U. S. Flare 908B squibs. It was necessary to use this ignition system because of monetary considerations.

The motor was static tested at the Corolla, North Carolina, facility on October 11, 1962. After initiation of the igniter, the motor failed to ignite for approximately 20 seconds. At that time, the motor over-pressurized, the case ruptured, and the nozzle was expelled. The nozzle was recovered intact and, except for a chipped graphite insert, showed no apparent damage. The ballistic recorders were inadvertently turned off approximately 10 seconds after igniter initiation. Consequently, it has been difficult to accurately determine the cause of over-pressurization. The motor had been stored in an uncontrolled atmosphere prior to static firing for a month and a half after radiographic inspection. Hence, it is possible that grain defects could have developed during this period as a result of temperature changes. It is suspected, however, that the gradual build-up of propellant gases during the long hang-fire situation created an explosive atmosphere which caused the motor to fail on ignition.

RECOMMENDATIONS FOR FUTURE TESTING

Further developmental testing of the integral nozzle closure and igniter would be required prior to the completion of the heavy-walled sub-scale motor test program. It is recommended that a series of preliminary

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ignition studies be conducted in small test motors containing only enough propellant to duplicate the actual propellant surface used in the 17-inch motor. After an igniter design was demonstrated to be satisfactory, a final heavy-walled steel, subscale spherical motor would be fired. The purpose of this firing would be to evaluate the lightweight nozzle cone as well as the final igniter design. The grain used in this test would be cast from the Arcane 42 aluminized propellant and would be modified so as to eliminate regressive burning characteristics. With the successful firing of this motor, the 17-inch subscale titanium motors could be fired to determine the ballistic performance of the beryllium-containing propellant.

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APPENDICES

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APPENDIX A
PROPELLANT FORMULATIONS AND INGREDIENTS

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PROPELLANT FORMULATIONS AND INGREDIENTS

Arcane 30 contains: 60 percent ammonium perchlorate (7:3 24-mesh unground/2TH 6900 rpm grind); 20 percent aluminum (Alcoa 123); and 20 percent JPL polyurethane binder¹.

Arcane 38 contains: 67.50 percent ammonium perchlorate (7:3 24-mesh unground/2TH 6900 rpm grind); 12.50 percent beryllium (Brush "17-micron"); and 20 percent JPL polyurethane binder.

Arcane 39 contains: 68.55 percent ammonium perchlorate (3:1 24-mesh unground/2TH 6900 rpm grind); 13.45 percent beryllium (Brush "17-micron"); and 18 percent JPL polyurethane binder.

Arcane 39A is the same as Arcane 39 except that the beryllium powder is Berylco "low-oxide".

Arcane 39AZ is the same as Arcane 39A except that the ammonium perchlorate is a 2:2:1 blend of unground 24-mesh; Pacific Engineering Corporation "-10+48 mesh"; and 2TH 6900 rpm grind.

Arcane 39Y is the same as Arcane 39 except that the ammonium perchlorate is a 5:2 blend of Pacific "-10+48 mesh" and Pacific "Class I Spherical".

Arcane 39Z is the same as Arcane 39 except that the ammonium perchlorate is a 2:2:1 blend of unground 24-mesh; Pacific "-10+48"; and 2TH 6900 rpm grind.

¹The "JPL polyurethane binder" referred to above is a polyurethane binder of the JPL X-535 type with 2,4-toluene diisocyanate, polypropylene glycol 2025-1, trimethylol propane, and Alrospers 11P in an equivalents ratio of 1.05/0.78/0.11/0.11. Nominal catalyst content is 0.472 percent ferric acetylacetonate. Neozone D (phenyl- β -naphthylamine) is used as an anti-oxidant in a concentration of 1.2 percent.

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Arcane 40 contains: 67.05 percent ammonium perchlorate (4:1 24-mesh unground/2TH 6900 rpm grind); 14.95 percent beryllium (Brush "17-micron"); and 18 percent JPL polyurethane binder.

Arcane 40A is the same as Arcane 40 except that the beryllium powder is Brush "325-mesh".

Arcane 40BX contains: 67.05 percent ammonium perchlorate (2:2:1 24-mesh unground/Pacific "-10+48 mesh"/2TH 6900 rpm grind); 14.95 percent beryllium (General Astrometals "400-mesh"); and 18 percent JPL polyurethane binder.

Arcane 40CX is the same as Arcane 40BX except that the beryllium powder is Brush "17 \pm 5 micron" (1755).

Arcane 40W is the same as Arcane 40 except that the ammonium perchlorate is a 3:2 blend of Pacific "-10+48 mesh" and Pacific "Class I Spherical".

Arcane 40X is the same as Arcane 40 except that the ammonium perchlorate is a 2:2:1 blend of unground 24-mesh; Pacific "-10+48 mesh"; and 2TH 6900 rpm grind.

Arcane 40Y is the same as Arcane 40 except that the ammonium perchlorate is a 5:2 blend of Pacific "-10+48 mesh" and Pacific "Class I Spherical".

Arcane 40Z is the same as Arcane 40 except that the ammonium perchlorate is a 5:3:1 blend of unground 24-mesh; American Potash and Chemical Co. "extra large"; and 2TH 6900 rpm grind.

Arcane 42 contains: 57.831 percent ammonium perchlorate (7:3 24-mesh unground/2TH 6900 rpm grind); 24.169 percent aluminum (Alcoa 123); and 18 percent JPL polyurethane binder.

Arcane 42X is the same as Arcane 42 except that the ammonium perchlorate is a 2:2:1 blend of unground 24-mesh; Pacific "-10+48 mesh"; and 2TH 6900 rpm grind.

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Arcane 42Z is the same as Arcane 42 except that the ammonium perchlorate is a 5:2 blend of Pacific "-10+48 mesh" and Pacific "Class I Spherical".

Arcane 47 contains: 67.83 percent ammonium perchlorate (2:2:1 unground 24-mesh/Pacific "-10+48 mesh"/2TH 6900 rpm grind); 14.17 percent beryllium (Brush "17-micron"); and 18 percent JPL polyurethane binder.

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Summary of Compositions and Theoretical Performance
of Arcane Formulations

<u>Formulations</u>	<u>Arcane 30</u>	<u>Arcane 38</u>	<u>Arcane 39</u>	<u>Arcane 40</u>	<u>Arcane 42</u>	<u>Arcane 47</u>
Composition, wt percent						
Binder	20.00	20.00	18.00	18.00	18.00	18.00
Beryllium	-	12.50	13.45	14.95	-	14.17
Aluminum	20.00	-	-	-	24.17	-
Ammonium Perchlorate	60.00	67.50	68.55	67.05	57.83	67.83
Flame Temperature, °K	3309	3394	3520	3556	3456	3540
Specific Impulse ^a , lb-sec/lb	260.7	280.9	281.2	282.4	261.1	281.9

Raw Materials and Suppliers

Polypropylene glycol 2025-one	Union Carbide, Baltimore, Maryland
Hylene T	DuPont, Wilmington, Delaware
Estane B-5720 (P-2 prepolymer).	B. F. Goodrich, Cleveland, Ohio
Ferric acetylacetonate	Norac, Azusa, California
Trimethylol propane	Celanese Corp., New York, N.Y.
Neozone D	DuPont, Wilmington, Delaware
Alrospere 11P	Geigy Corp., Yonkers, New York
Diocetyl azelate	Emery Industries, Cincinnati, Ohio
Beryllium powder	Brush Beryllium Corp., Cleveland, Ohio (BBC)
	Beryllium Corporation of America, Hazelton, Pa. (Berylco)
	General Astrometals, Yonkers, N.Y.
Aluminum powder (Alcoa)	Alcoa, Conshohocken, Pennsylvania

^aSpecific impulse for a chamber pressure of 1000 psi, an exhaust pressure of 1 atmosphere, and pre-combustion temperature of 20°C, assuming shifting chemical, thermal, and momentum equilibria throughout expansion of the combustion product through the rocket nozzle.

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Raw Materials and Suppliers (continued)

Ammonium perchlorate	American Potash, New York, N.Y. Pacific Engineering, Henderson, Nevada
DB castor oil	Baker Chemical Co., Philipsburg, New Jersey
Hylene M	DuPont, Wilmington, Delaware
Polyethylene glycol 400	Union Carbide, Baltimore, Maryland
LP-3	Thiokol Chemical Co., Trenton, New Jersey

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APPENDIX B

FACILITIES FOR PROCESSING AND TESTING BERYLLIUM PROPELLANT

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FACILITIES FOR PROCESSING AND TESTING BERYLLIUM PROPELLANT

Under the sponsorship of the Air Force and the Advanced Research Projects Agency, Atlantic Research Corporation has evaluated powdered beryllium metal as a high-energy fuel in solid propellants by the fabrication and static firing of both 10-pound and 50-pound test grains. Because of the high toxicity of beryllium and its compounds, this work has been carried out in an isolated propellant-development facility especially designed and constructed for the purpose. The avoidance of a neighborhood contamination above that allowed under Atomic Energy Commission recommended standards has required particular effort in the selection of equipment and methods for static-firing tests. This section discusses the facilities and specialized equipment that have resulted in the accomplishment of static-firing tests within the stringent requirements recommended by the Atomic Energy Commission. The industrial hygiene and air-pollution control program in operation is described in Appendix E.

The high-energy propellant facilities utilize an 80-acre portion of the Atlantic Research Pine Ridge Plant (Figure B-1). This plant occupies a 588-acre tract in rural Prince William County, Virginia, approximately 35 miles west of Washington, D. C. The terrain is gently rolling and heavily wooded. Annual rainfall averages over 30 inches and is well distributed throughout the year. Wind velocities are usually moderate, and periods of complete calm are rare. Prevailing winds throughout most of the year are from such directions that areas downwind from the project are largely uninhabited for distances up to several miles.

These characteristics combine to give the selected site favorable conditions for atmospheric dilution of stack losses, low "dustiness" or potential for resuspension of any sedimented dusts or fumes, and an acceptably low density of neighbors who might be exposed to toxic clouds in the event of a large-scale accident.

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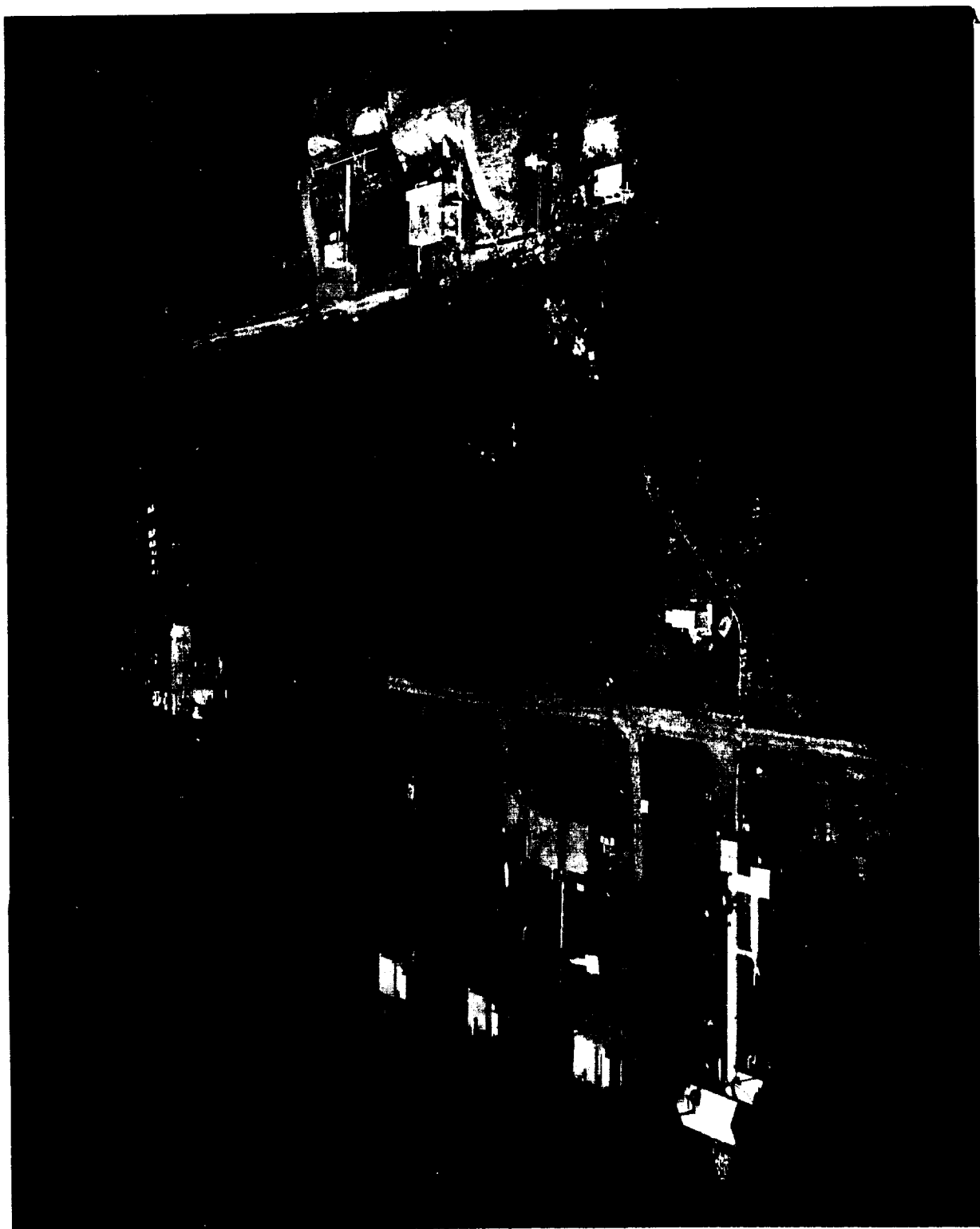


Figure B-1. High-Energy Propellant Facilities.

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Except for the problems of industrial hygiene peculiar to working with powdered beryllium, all propellant processing is straightforward. Operations during which beryllium might become airborne are conducted in a two-man glove box. Propellant mixing is accomplished with a planetary mixer fitted with a vacuum cover. This equipment is shown in Figure B-2. Motor casting is carried out by slit-filling of the propellant into an evacuated motor. A mixer and vacuum casting chamber are connected for direct casting from the mixer. A forced convection, hot-air oven, provided with controlled-temperature hot water in finned-tube heating coils is used for propellant curing operations. The cured grains are trimmed in the two-man glove box prior to assembly of the motors.

To conduct safe static-firing tests of beryllium propellants and avoid contamination of both the immediate vicinity and surrounding areas farther downwind, it is necessary to cleanse rocket exhaust gases of toxic materials before release into the atmosphere. At Atlantic Research this is accomplished by firing the rocket motor into a closed chamber and venting the fumes through a Pease-Anthony venturi scrubber which removes particulate matter from the gases before discharge to the atmosphere through a stack. The following paragraphs discuss the operation of this facility.

Static firing of beryllium-containing solid-propellant grains are conducted in the large test tunnel shown in Figure B-3. This tunnel, with a volume of approximately 7,000 cubic feet is composed of a 1-inch-thick steel pressure vessel 8 feet in inside diameter and 140 feet long. An internal dished head with a 20-inch manhole-flange divides the tunnel into two parts: a 14-foot-long loading and decontamination chamber, and a firing chamber. The Pease-Anthony scrubber is connected to the tunnel at the downstream end.

The thrust stand consists of two parallel steel rods, mounted horizontally to a 3-inch-thick steel blind-flange that is fitted to the 20-inch manhole-flange of the firing chamber. The rocket motor is mounted on a carriage which rides on the horizontal steel rods by means of linear roller bearings. The load cell is mounted directly to the steel blind-flange and attached

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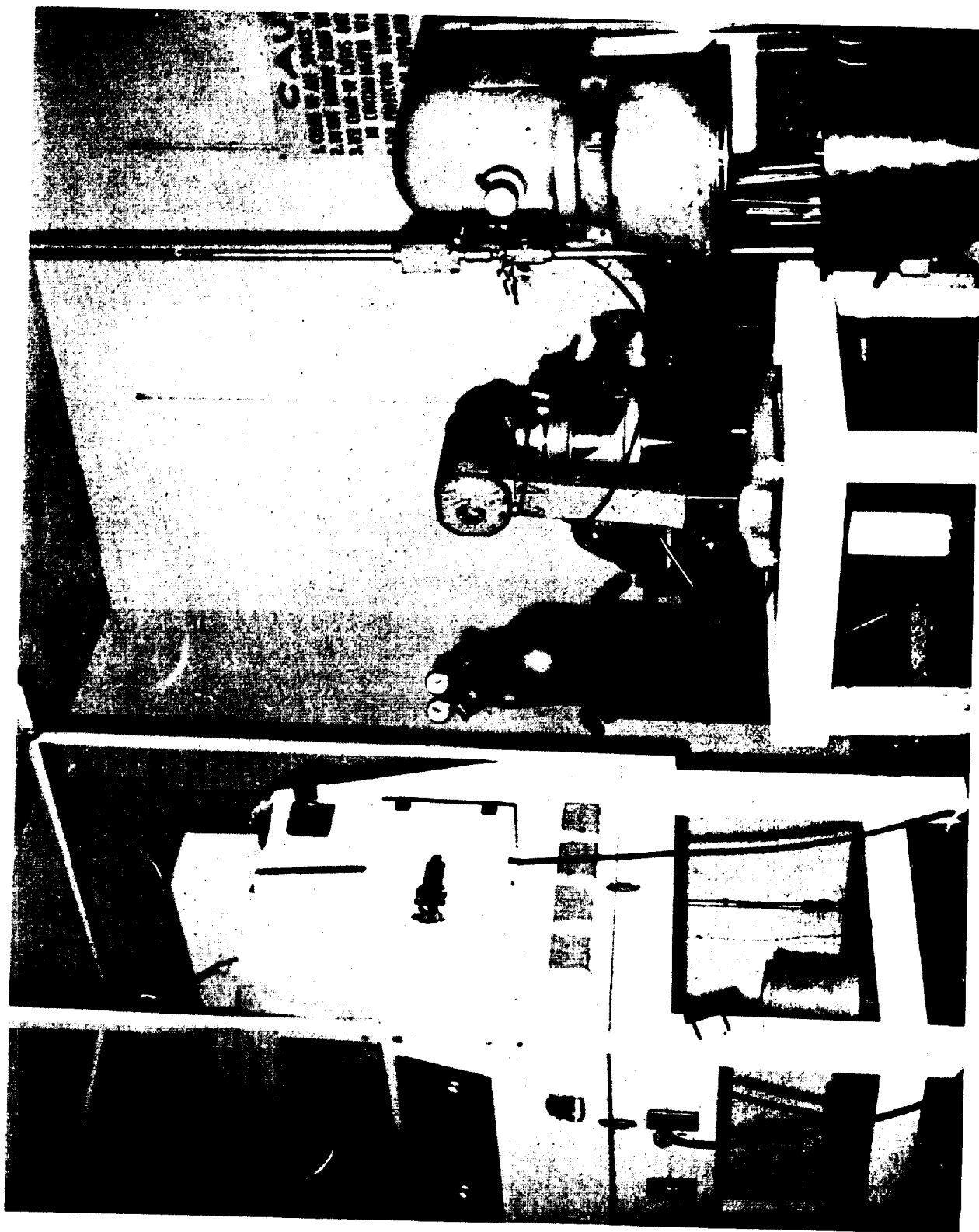


Figure B-2. Propellant Mixing Facilities.

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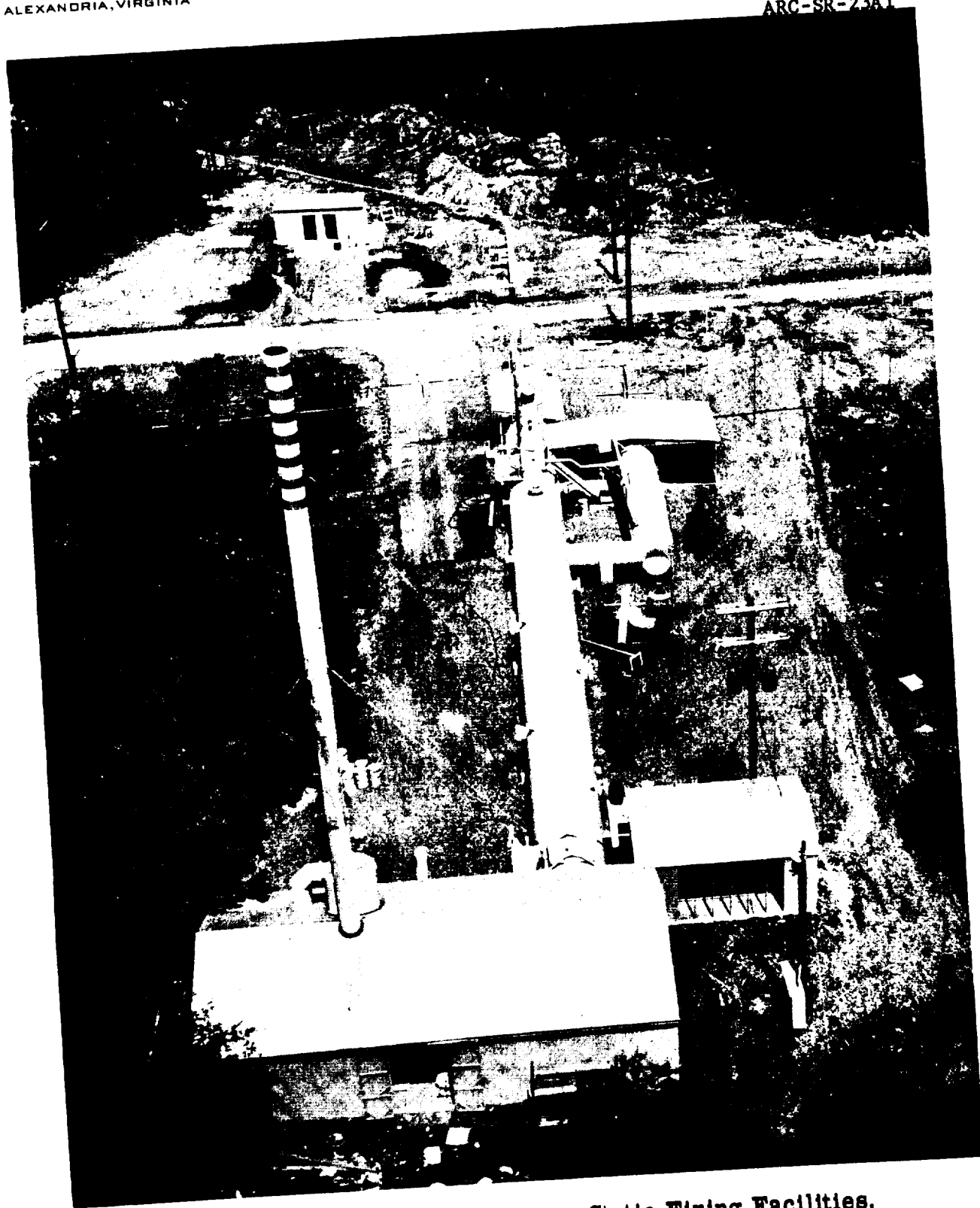


Figure B-3. Controlled Atmosphere Static Firing Facilities.

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to the rocket motor through a mono-axial thrust bearing. Alignment of the rocket motor and load cell is checked periodically. Instrumentation leads are brought through sealed connectors on the steel blind-flange.

To prevent the instantaneous rate of gas flow into the scrubber from exceeding design limits, there is a valve between the tunnel and the scrubber. This valve is closed during the firing, and the exhaust gases are completely contained. After the firing the gases are slowly bled into the scrubber at a rate such that the scrubber efficiency remains high. To minimize tunnel pressure buildup, a water-deluge system is used to cool the rocket motor exhaust gases during the static firing. The water-deluge tank is on top of the firing tunnel. The deluge tank is charged with about 600 gallons of water, and the space above the water is pressurized to 300 psig. Upon opening of a remotely controlled valve at the bottom of the deluge tank at the moment of firing, the water flows through a distribution pipe into the rocket exhaust stream in a time interval set (by means of a manual valve) to coincide approximately with the firing duration of the rocket motor. In firings of 10-pound grains, the tunnel pressure buildup is normally about 2 psi. In 50-pound motor firings, this pressure buildup is normally about 7 psi. To simulate firings at sea level conditions, the firing tunnel is normally evacuated slightly prior to firing using the large vacuum pump discussed subsequently in connection with simulated altitude firings. By this means, the average tunnel pressure during 10-pound motor firings is maintained at approximately sea level conditions.

A digital instrumentation system is used for thrust measurements, while analog records are obtained for both thrust and pressure (Figure B-4). Thrust is measured with an Alinco Model 341D load cell, rocket motor pressure with an Alinco Model 311 gage, and tunnel pressure and diffuser tube pressure with Statham absolute pressure gages, ranges of 0 to 2, 0 to 5, and 0 to 25 psia. Precision calibrating resistors are used with a 6-wire circuitry. The values of the calibrating resistors corresponding to pounds-force for each load cell or to psi for each pressure transducer are obtained through direct calibration against National Bureau of Standards calibrated

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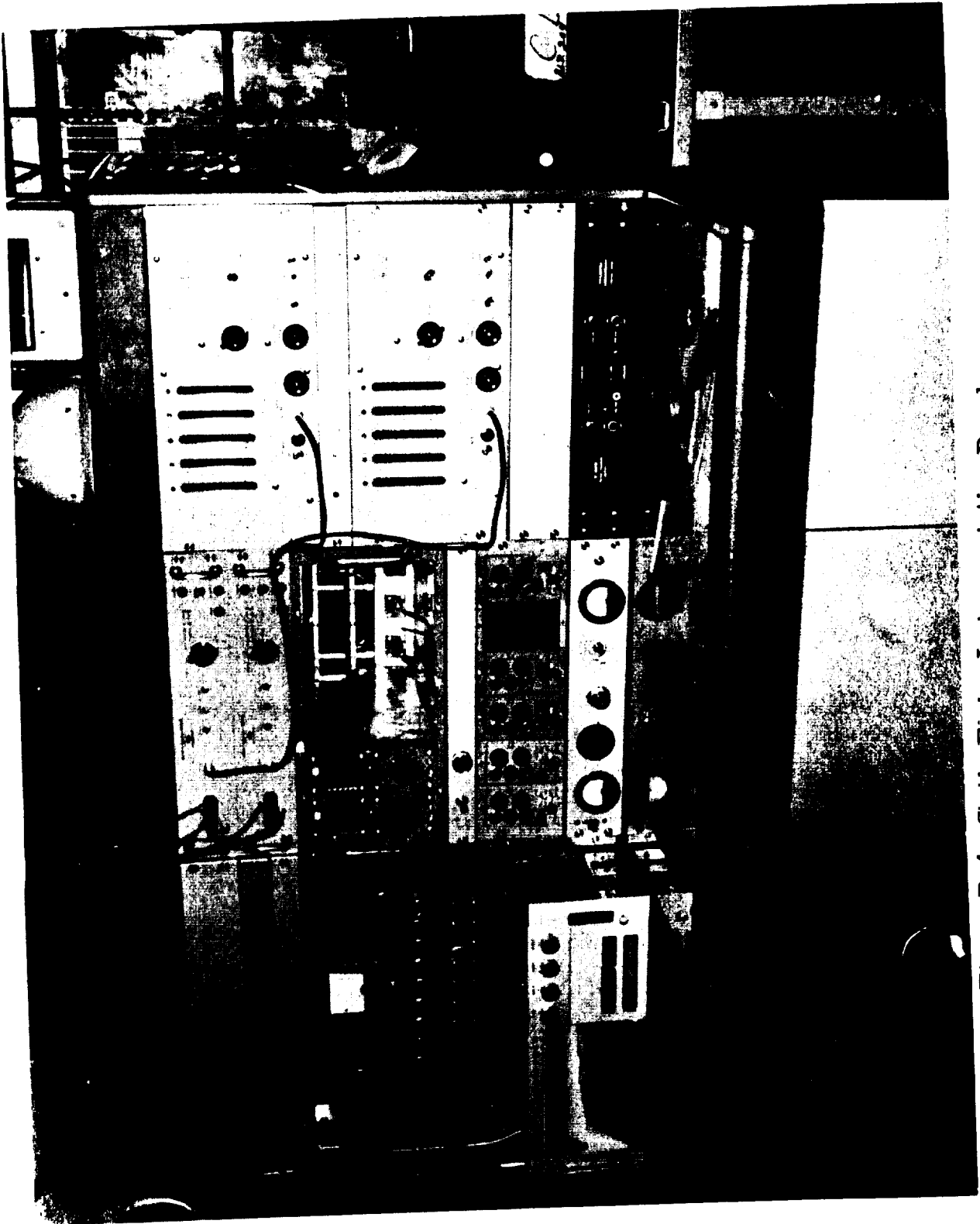


Figure B-4. Static Firing Instrumentation Panels.

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weights. The gages are recalibrated at routine intervals. Immediately before and after each firing, the calibration resistors are switched into the circuit to provide reference points. Four calibration values are normally used corresponding to 25, 50, 75, and 100 percent of full scale for the range of the particular gage. For vacuum firings, pre-firing conditions are obtained with manometers or McLeod gages. To prevent pressure fluctuations in the tunnel from affecting the output of the load cells, vented load cells are used.

The 150-pound firings were carried out at Atlantic Research Corporation's North Carolina site. Known as Corolla, the site is on the Outer Banks roughly midway between Virginia Beach, Virginia, and Nags Head, North Carolina. The location is indicated on the area map in Figure B-5.

Corolla stretches for approximately five miles along the Outer Banks. It is accessible by vehicle, boat, and small plane and is served by electric power and telephone. The terrain is rolling, seashore dunes largely covered by grass and trees, punctuated at one-half to one mile intervals by giant dunes.

Static firing of beryllium-containing, solid-propellant grains at the Corolla, North Carolina, site were conducted in open air using prevailing land to sea breezes to disperse rocket exhaust gases and prevent contamination of land area. A 12-foot-square concrete pad supports a vertical thrust stand consisting of a 2-inch steel base plate 42 inches square with a load-cell mounting hole tapped in the center. Three vertical "eye" beams are located equidistant from the center on a 20-inch-diameter circle 120 degrees apart. Flexure mounts placed atop these beams supported the flexures which in turn prevent the motor from turning in the mount. The motor is fastened in a harness which connects to a mono-axial thrust bearing mounted on the load cell.

For simulated altitude firings a diffuser tube is placed over the thrust stand and sealed. The tube is pumped down to the desired initial vacuum by means of a Kinney K-8 vacuum pump.

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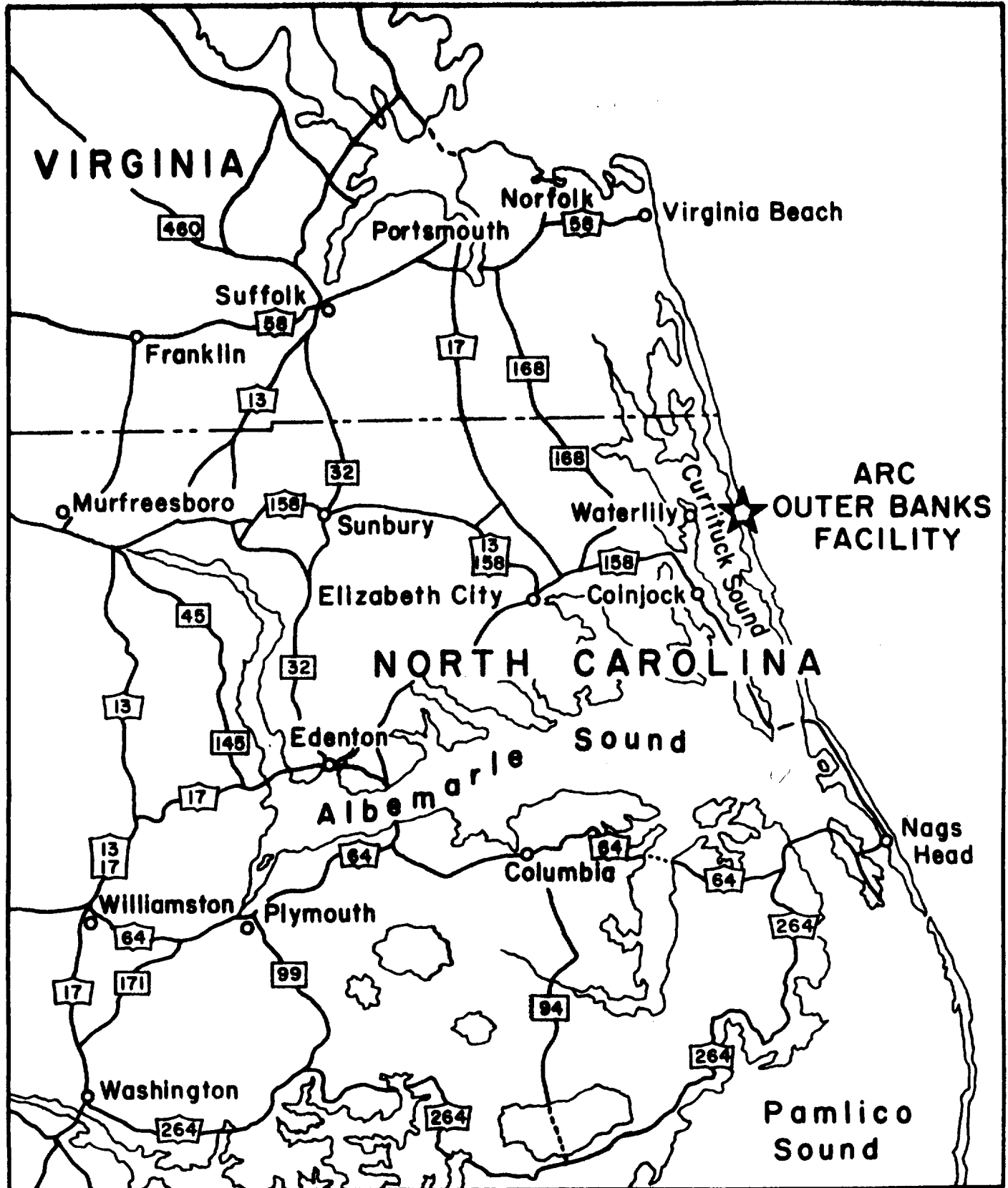


Figure B-5. Regional Map of Outer Banks Facility Area.

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A digital instrumentation system is used for thrust and pressure measurements. Analog records are obtained as well.

Thrust is measured by means of an Alinco 341D Vented Load Cell. Pressure is measured by means of an Alinco 311 or 151-C1 Pressure Transducer. Each channel is in circuit with an SRB 200F Video Instrument Power Supply, CEC Model AI 233B Amplifier, Dymec Model 2211 AR Voltage to Frequency Converter, and a Hewlett Packard Model 521CR Counter. Analog records are recorded on a Heiland Visicorder.

Diffuser pressure is recorded on analog from 0 to 5 and 0 to 25 Statham Absolute Pressure Gages.

Calibrations are carried out as previously described.

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APPENDIX C
STANDARD OPERATING PROCEDURES FOR
ASSEMBLY OF 17-INCH SPHERICAL MOTOR

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ARC-SR-23A1

STANDARD OPERATING PROCEDURES FOR
ASSEMBLY OF 17-INCH SPHERICAL MOTOR

Reference: ARC drawings - T6-23914, ED-04-09-38-1, and TD04-09-21-6

A. MOTOR PREPARATION

1. Weight of bare motor is obtained (300 pound scales¹ - 3 separate weighings for this and subsequent weighings).
2. Weight obtained after motor is lined with V-44 rubber asbestos.
3. Polyurethane foamed in place and machined. Slots cut in foam for fins. Motor weighed.
4. Two coats of Stanley primer brushed on V-44 to intersection with foam (excluding foam). Primer is a mixture of two parts 40X-415 Stanley primer and one part 79R-192 Stanley thinner, both manufactured by Stanley Chemical Co. The first coat is allowed to air dry for 1 hour. The second coat is applied and cured 2 hours at 50°C.
5. Two coats of PUX-251² are brushed onto Stanley primer (excluding foam). Two hundred grams will be used for each coat. The first coat will be allowed to cure at ambient temperature for a minimum of 4 hours (preferably overnight) before application of the second coat. The PUX is mixed in the order given in the formulation, then degassed with stirring for 5 minutes. The motor is then weighed to obtain weight of Stanley primer and PUX-251. An argon atmosphere is maintained in motor.

¹Fairbanks-Morse. Weight to nearest 0.01 pound.

²Formulation:

PEG 400	9.95%	Araldite CN-502	4.98%
LP-3	9.95	Hylene M	29.81
		D.B. Castor Oil	45.31

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6. To prevent bond of propellant to foam, a coat of epoxy (90 parts Araldite CN-502 to 10 parts Araldite HN-951 hardener) is brushed on all exposed parts of foam and allowed to cure at ambient temperature.
7. Final weight of motor obtained.
8. Collar and fins are installed; motor is now ready for casting.

B. CASTING PROCEDURE

1. Motor is placed in vacuum chamber. Funnel is set up with 3 inch diameter, 0.060-inch slits or teflon disc. A rubber stopper with brass rod attached is placed over slits and chamber evacuated to 29-30 inches Hg.
2. Propellant is poured into funnel and rubber stopper removed. A blanket of argon is maintained on propellant in funnel at all times. The level of the propellant in funnel is closely watched and maintained at a depth no less than 3 inches to prevent blow-through and loss of vacuum.
3. The motor is filled to a depth measuring 2-5/8 inches below the inner edge of the V-44 at the collar.
4. When it appears that this depth is reached, the flow of propellant is stopped by inserting the rubber stopper on top of the slits and the vacuum broken with argon. If a measurement shows more propellant is needed, chamber is evacuated (29-30 in Hg) and more propellant added. This procedure is followed until correct depth is obtained.
5. The mandrel is assembled (Drawing T6-23914) and lowered (manually) slowly into the propellant until the spider can be locked into place on the collar.

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ARC-SR-23A1

C. GRAIN CURING

1. The grain is cured at 135°F for 120 hours. A record of the cure cycle is obtained with a Thermo-Electric potentiometer type recorder.
2. After curing, the grain is cooled overnight to ambient temperature.

D. GRAIN STRIPPING AND TRIMMING

1. The motor is positioned and attached to the stripping stand in the cure building, and the mandrel pulled remotely with a Black Hawk hydraulic unit.
2. The collar is removed, the web of propellant between the fins and core is cut away and the fins removed.
3. Any propellant along the top edge of the foam is trimmed away (according to Drawing ED-04-09-38-1).
4. The slots in the beveled edge of V-44 are filled with cured V-44 bonded with Goodyear Pliobond.
5. A loaded motor weight is obtained.
6. The foam is cut away exposing the pressure tap(s).
7. Motor re-weighed.

E. FINAL ASSEMBLY

1. The nozzle and igniter is assembled (Drawing ED 04-09-38-1).
2. The nozzle, without O-ring, is lowered into position to check fit and then extracted.
3. The O-ring is installed and coated with Dow-Corning Vacuum grease.
4. The nozzle is installed and each nut torqued to 150 in/lb.

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APPENDIX D
DESIGN AND STRESS REPORT

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BY: J. H.
CHK: _____

J.P.L. MOTOR

DATE
8/16/62
PAGE 1
OF 45

JET PROPULSION LABORATORY

SPHERICAL MOTOR

DESIGN AND STRESS

REPORT

MECHANICAL ENGINEERING DIVISION

REPORT NUMBER SR207

PREPARED BY:

CHECKED BY:

J. H. H. H.
C. E. H. H.



BY: _____
CHK: _____

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INTRODUCTION

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Data for the minimum weight design of the 36 inch spherical case is derived from the design, fabrication, and hydrostatic test of the 17 inch experimental spherical case.

The wall thickness "t" of the spherical part of the case is given unambiguously by the conventional;

$$t = \frac{PR}{2\sigma_M}$$

Where σ_M = Membrane Design Stress.

For weight considerations then it is desirable to reduce to a minimum the flange profile, whose dimensions primarily depend on screw thread diameter and thread length. Studs are chosen in preference to removeable bolts because of further possible reduction in flange length.

The stud diameter is determined by producibility of a minimum diameter tapped hole in the Titanium flange.

The internal bending moment (M_o) and shear force (Q_o) at the geometrical discontinuity of the sphere - flange joint are found by the method of consistency of deformations.

The stress limit (σ_L) requires a local increase in the shell thickness (t_o) such that,

$$\frac{PR}{2t_o} + \frac{6M_o}{t_o^2} \leq \sigma_L$$

The edge moment (M_o) decays along the meridian as

$$M_w = M_o \exp(-\lambda w)$$

with good approximation where w is the meridional angle from the edge of the joint and λ is the shell parameter.

$$\lambda = \left[3(1-\nu^2) \frac{R^2}{t^2} \right]^{1/4}$$

Accordingly, the w is found so that

$$\frac{PR}{2t} + \frac{6M_w}{t^2} = \sigma_L$$



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where t is the basic shell membrane thickness.

The motor is to operate effectively at a short time elevated temperature (150°F) condition. Therefore, the following strengths are required of the case material.

Material: 6AL-4V Titanium Alloy

Min. Tensile Strength at room temperature = 170,000 PSI

Min. Yield Strength at room temperature = 165,000 PSI

Min. Yield Strength at 150°F = (Short Time Duration) 155,000 PSI

The design stresses are; $\sigma_M = 150,000$
 $\sigma_L = 155,000$.

The motor maximum operating pressure is 800 PSI.

The design pressure is 1000 PSI.

So the minimum margin of safety = .25

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WALL THICKNESS

$$\sigma_M = \frac{PR}{2t}$$

σ_M = Membrane Design Stress

P = Design Pressure

R = Spherical Radius

t = Wall Thickness

$$\sigma_M = 150,000 \text{ psi (Maximum)}$$

$$P = 1,000 \text{ psi}$$

$$R = 18 \text{ in}$$

$$t = \frac{1000 \times 18}{2 \times 150,000} = .060 \text{ in}$$

Specification of Wall Thickness is:

$$t = .060 \begin{matrix} -.000 \\ +.003 \end{matrix}$$



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ANALYSIS OF STUDS

Force on studs

$$F = \frac{P \pi D^2}{4} = \frac{1000 \times 3.14 \times 16^2}{4}$$

D = Stud bolt circle diameter

F = 201,000 lbs.

Force per stud using 36-(1/4") studs

$$f = \frac{201,000}{36} = 5600 \text{ lbs/stud}$$

Stress on studs

$$\sigma = \frac{F}{A} = \frac{4 \times 5600}{3.14 \times (.2062)^2} = 168,000 \text{ psi}$$

$$A = \frac{\pi d^2}{4}$$

d = Minor diameter of thread.

Specification:

36-1/4" studs made of 416 stainless steel heat treated to $\sigma_{ult} = 190,000$.

$$M.S. = \frac{190,000}{168,000} - 1 = .13$$

Stainless steel is chosen for its compatability with case material.

Stress on threads

$$\frac{f}{\pi (P.D.) (n-2) t} \leq \frac{\sigma_{ult}}{4} \leq 40,000 \text{ (Titanium)}$$

t = Thread thickness at P.D.

P.D. = Pitch Diameter.

n = Number of threads.



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Say 2 Inactive threads.

$$n-2 = \frac{3600}{3.14 \times .228 \times .019 \times 40,000} = 10.2 \text{ Threads}$$

n = 13 Threads.

Using 28 threads per inch, there are 14 threads in .500 inches.

MINIMUM FLANGE PROFILE FOR ASSEMBLY FUNCTION

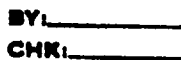
The minimum flange length, determined by stud thread length, is 0.50 inch.

The flange face should be two times the stud diameter. So the face is 0.50 inch.

The mandrel size determines the size of the inside diameter of the flange as scaled up from the 17" motor

$$d = \frac{17.0}{36} \times 7.062 + .708$$

$$d = 13.66 \text{ in.}$$

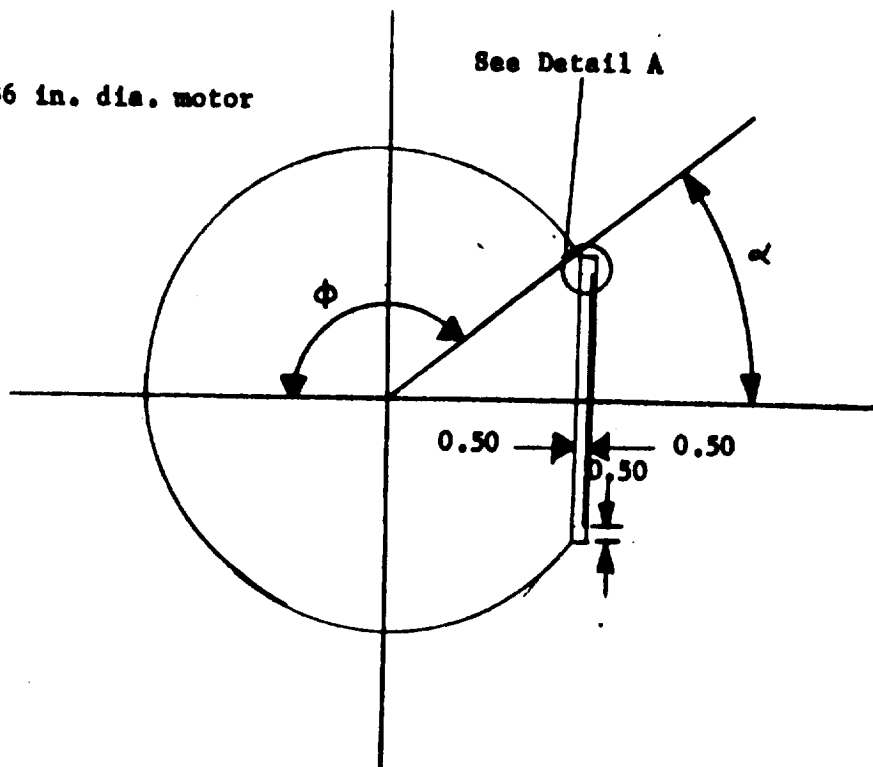


MOTOR CASE

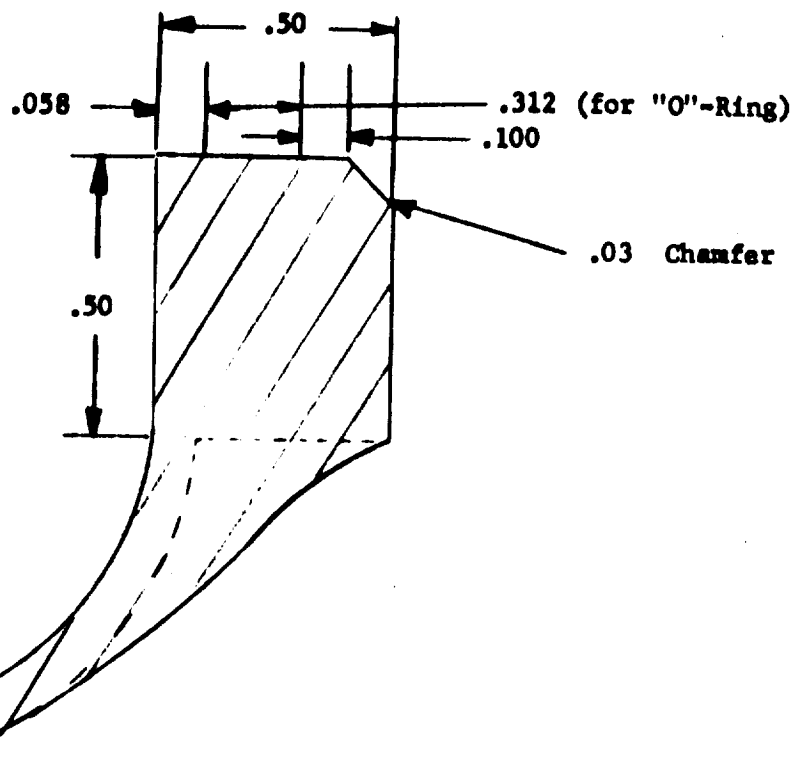
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FLANGE PROFILE

- See Detail A**



- 2.**



DETAIL A



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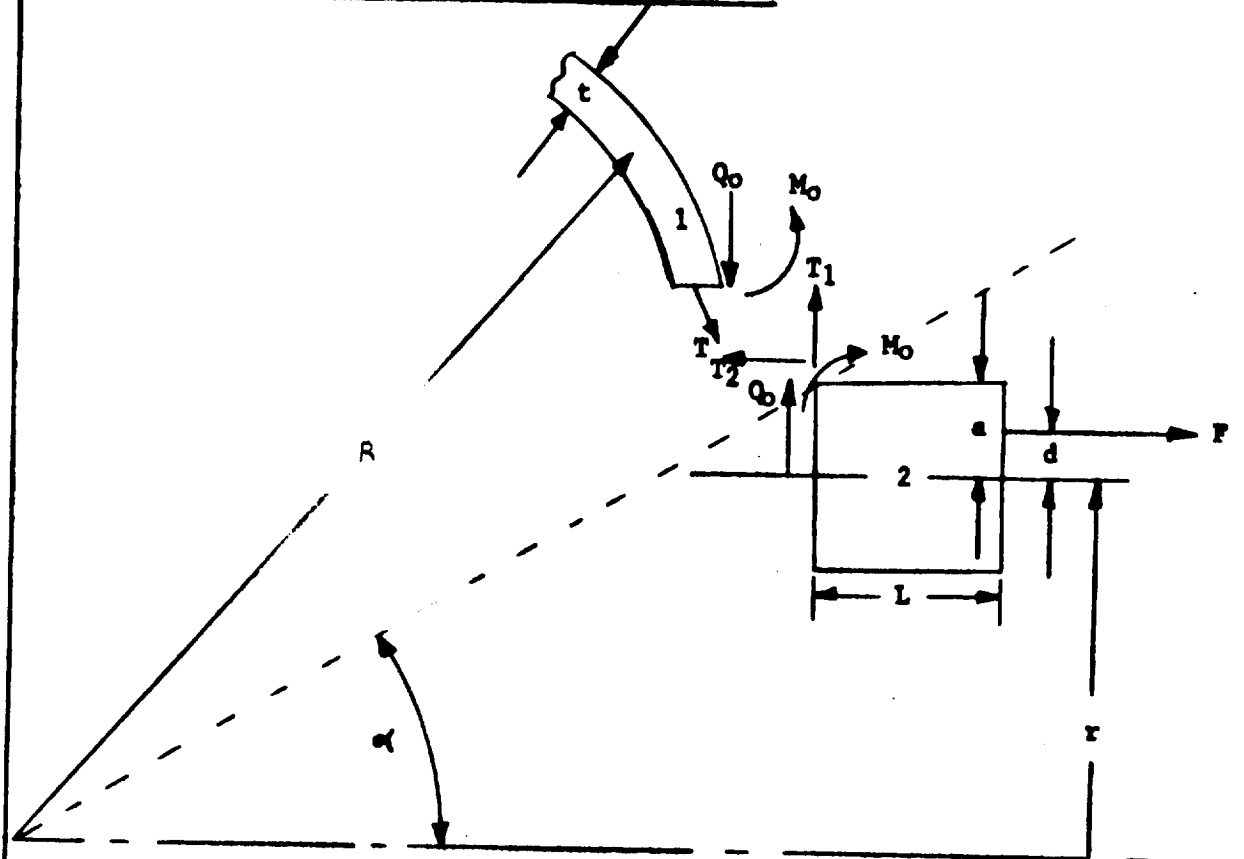
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ANALYSIS OF JOINT - SPHERE TO FLANGE



$$W_1 = W_2$$

$$-W_{1p} + \delta_{1Q} \cdot Q_0 - \delta_{1M} \cdot M_0 =$$

$$-W_{2p} - \delta_{2Q} \cdot Q_0 - \delta_{2M} \cdot M_0 - \delta_{2Q} \cdot T_1 + \delta_{2M} (aT_2 - Fd)$$

$$\theta_1 = \theta_2$$

$$\theta_{1Q} \cdot Q_0 + \theta_{1M} \cdot M_0 = \theta_{2Q} \cdot Q_0 - \theta_{2M} \cdot M_0 - \theta_{2Q} \cdot T_1 +$$

$$\theta_{2M} (aT_2 - F \cdot d)$$



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The stud eccentricity (d) with the centerline of the flange can be varied somewhat and can be chosen to reduce the edge moment (Mo).

$$\text{Say } - .0625 \text{ in. } \leq d \leq +.0625$$

with positive d as shown in the sketch. An investigation shows that with d chosen as .0625 the minimum edge moment (Mo) occurs. (Ref. page 13)

$$T = \frac{PR}{2} = \frac{1000 \times 18}{2} = 9000 \text{ \#/in.}$$

$$T_1 = T \cdot \cos \alpha = 7983$$

$$T_2 = T \cdot \sin \alpha = 9000 \times .462 = 4156 \text{ \#/in.}$$

$$aT_2 = .25 \times 4156 = 1078 \text{ \#/in}$$

$$P \cdot d = 235 \text{ \#/in/in.}$$

$$\nu = .30$$

$$\alpha = \text{Joint Angle}$$

$$\alpha = \sin^{-1} \frac{1666}{36} = 27.5^\circ$$

SPHERICAL COEFFICIENTS*

$$R = 18.0 \text{ in.}$$

$$t = .06 \text{ in.}$$

$$\phi = 180^\circ - \alpha = 152.50$$

$$\sin \phi = .46175$$

$$\cot \phi = -1.921$$

$$\cos \phi = .88701$$

$$\lambda = 1.285 \sqrt{\frac{R}{t}} = 22.26$$

$$K_1 = 1 + \frac{(1-2\nu)}{2\lambda} \cot \phi$$

$$K_1 = 1.017$$

$$K_2 = 1 + \frac{(1+2\nu)}{2\lambda} \cot \phi$$

$$K_2 = 1.069$$

$$\delta_{1Q} = \frac{\lambda R \sin^2 \phi (K_2 + 1/K_1)}{Et} = \frac{22.26 \times 18 \times .46175^2 (1.017 + .935)}{E \times .06}$$

$$\delta_{1Q} = \frac{2780}{E}$$

$$\delta_{1M} = \frac{2\lambda^2 \sin \phi}{Et K_1} = \frac{2 \times 22.26^2 \times .46175}{E \times .06 \times 1.017} = \frac{7499}{E}$$

$$\theta_{1Q} = \delta_{1M}$$

* Reference: ROARK Page 272



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$$\theta_{1M} = \frac{4 \lambda^3}{E t R K_1} = \frac{4 \times 22.26^3}{E .06 \times 18 \times 1.017} = \frac{40,169}{E}$$

$$W_{1p} = \frac{.35 PR^2 \sin \phi}{E t} = \frac{.35 \times 1000 \times 18^2 \times .46175}{E \times .06}$$

$$W_{1p} = \frac{872,700}{E}$$

SHORT CYLINDER COEFFICIENTS *

$$r = 8.08$$

$$B = \frac{1.285}{\sqrt{2ar}} = .6393$$

$$2a = .5$$

$$BL = .3197$$

$$L = .5$$

$$\frac{1}{D} = \frac{87.36}{E}$$

$$D = \frac{Et^3}{12(1-\nu^2)}$$

$$\delta_{2Q} = \frac{1}{BL} \cdot \frac{1}{B^3 D} = \frac{87.36}{.3197 \times E \times .6393^3} = \frac{1046}{E}$$

$$\delta_{2M} = \frac{1}{(BL)^2} \times \frac{1.5}{B^2 D} = \frac{1.5 \times 87.36}{.3197^2 E .6393^2} = \frac{3137}{E}$$

$$\theta_{2Q} = \delta_{2M}$$

$$\theta_{2M} = \frac{1}{(BL)^3} \times \frac{3}{BD} = \frac{1}{.3197^3} \times \frac{87.36 \times 3}{E .6393}$$

$$\theta_{2M} = \frac{12,540}{E}$$

$$W_{2p} = \frac{.85 PR^2}{E (2a)} = \frac{.85 \times 1000 \times 8.08^2}{E .5}$$

$$W_{2p} = \frac{110,986}{E}$$

* Reference: NAMA TR E-103



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$$(\delta_{1Q} + \delta_{2Q}) Q_0 + (\delta_{2M} - \delta_{1M}) M_0 = W_{1p} - W_{2p} + \delta_{2M} (aT_2 - F \cdot d) - \delta_{2Q} \cdot T_1$$

$$(\sigma_{RQ} - \sigma_{1Q}) Q_0 + (\sigma_{1M} + \sigma_{2M}) M_0 = \sigma_{2M} (aT_2 - F \cdot d) - \sigma_{2Q} \cdot T_1$$

$$3826 Q_0 - 4362 M_0 = 1.957 \times 10^6$$

$$-4362 Q_0 + 52711 M_0 = 9.125 \times 10^6$$

$$Q_0 - 1.140 M_0 = 511.5$$

$$-Q_0 + 12.084 M_0 = 2091.93$$

$$10.944 M_0 = 2603.43$$

$$M_0 = 237.9$$

$$Q_0 = 511.5 + 271.2$$

$$Q_0 = 782.7$$

$\frac{1}{E}$ is contained in every term of both equations and is divided out.

t_0 = Actual spherical thickness at joint

$$\frac{PR}{2t_0} + \frac{6M_0}{t_0^2} = 155,000$$

$$\frac{9000}{t_0} + \frac{1428}{t_0^2} = 155,000$$

$$155 t_0^2 - 9 t_0 - 1.428 = 0$$

$$t_0 = \frac{-9 \pm \sqrt{81 + 855}}{310}$$



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$$t_0 = .13 \text{ in}$$

$$M_w = M_0 \exp(-\lambda w)$$

$$\exp(-\lambda w) = \frac{M_w}{M_0} = \frac{3}{238} = .0126$$

$$\lambda w = 4.37$$

$$w = \frac{4.37}{22.26} = .197 \text{ RAD}$$

$$w = 11.3^\circ$$



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CHECKS ON ANALYSIS

A check on the method of analysis and on the design criteria is provided by the stress results obtained from the hydrostatic pressure test of the 17 inch experimental case. These results are shown in the Atlantic Research Corporation Stress Results Report (SR 205). The predicted stresses compare quite favorably with the measured stresses at 1000 psi pressure.

Another check on the applicability of the method was provided by an analysis of data from a destructive pressure test on a 4 inch spherical head at the Airite Corporation. The Vessel was analyzed using the same theory on the thin wall joint. The failure occurred exactly where the theory located maximum moments because of edge shears and moments away from the joint.



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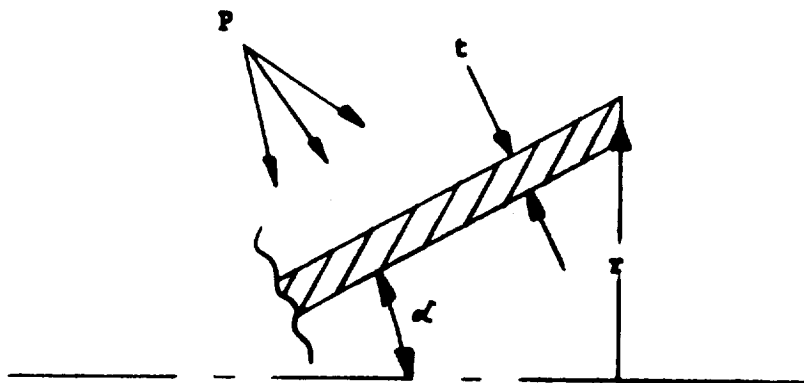
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INTERNAL EXPANSION CONE



$$\sigma_M = \frac{Pr}{t \cos \alpha} *$$

σ_M = Membrane Stress
P = Pressure
t = Wall Thickness

Material: 150 RPD

$$\sigma_{ult} = 26,000 \text{ in compression } **$$

$$\sigma_M = 20,000$$

$$t = \frac{1000 \times 7.83}{20,000 \times .94}$$

$$t = .416 \text{ in}$$

$$M.S. = \frac{26000}{20000} = 1.3$$

$$\sigma_{ult} \text{ (in tension) } *** = 6850 \text{ psi (Min.)}$$

Minimum value of α for nozzle used for calculating t.

** Ref. Raybestos - Manhattan, Inc.

* Ref. Roark Page 268



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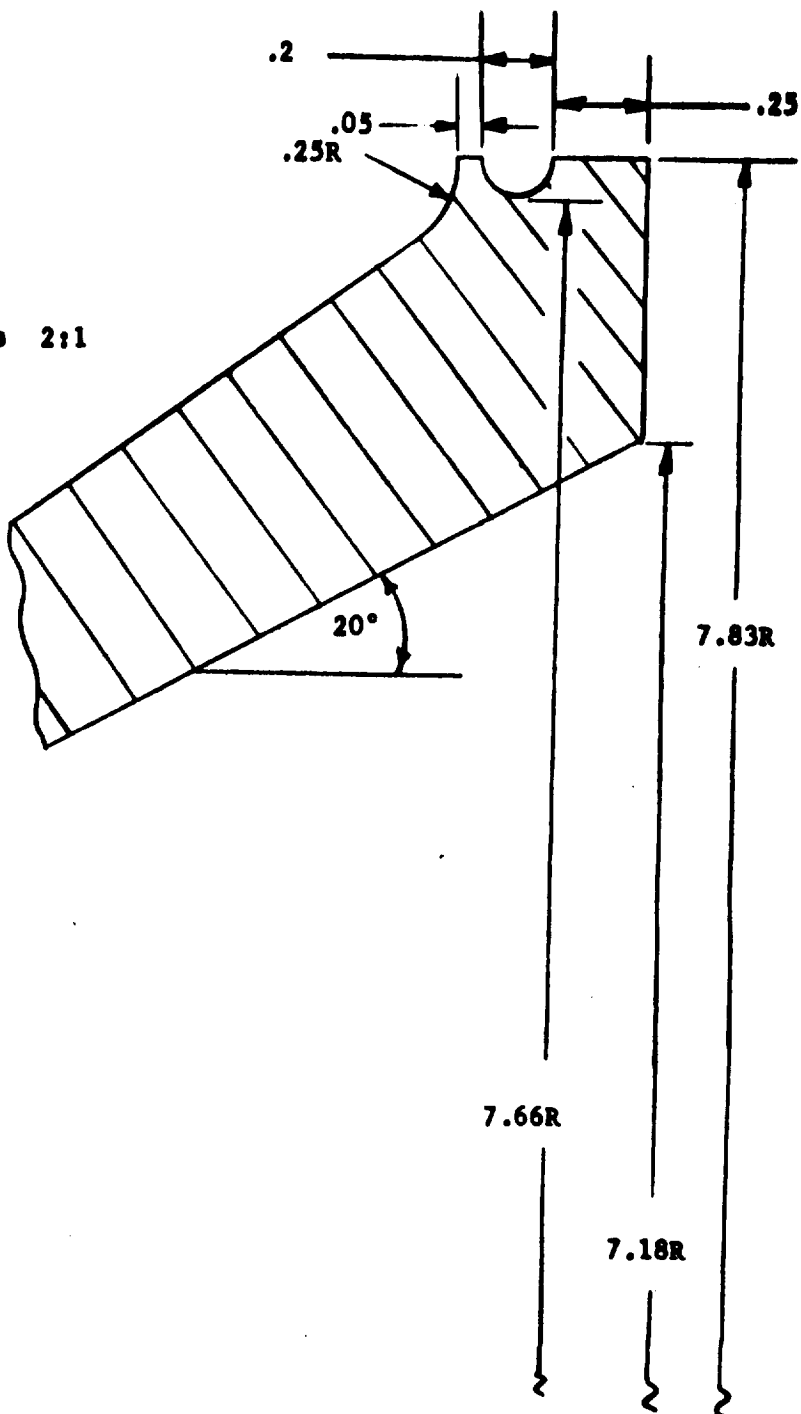
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DISCONTINUITY ANALYSIS OF LARGE END OF INTERNAL EXPANSION CONE.

Approximate scale is 2:1



ACTUAL DIMENSIONS

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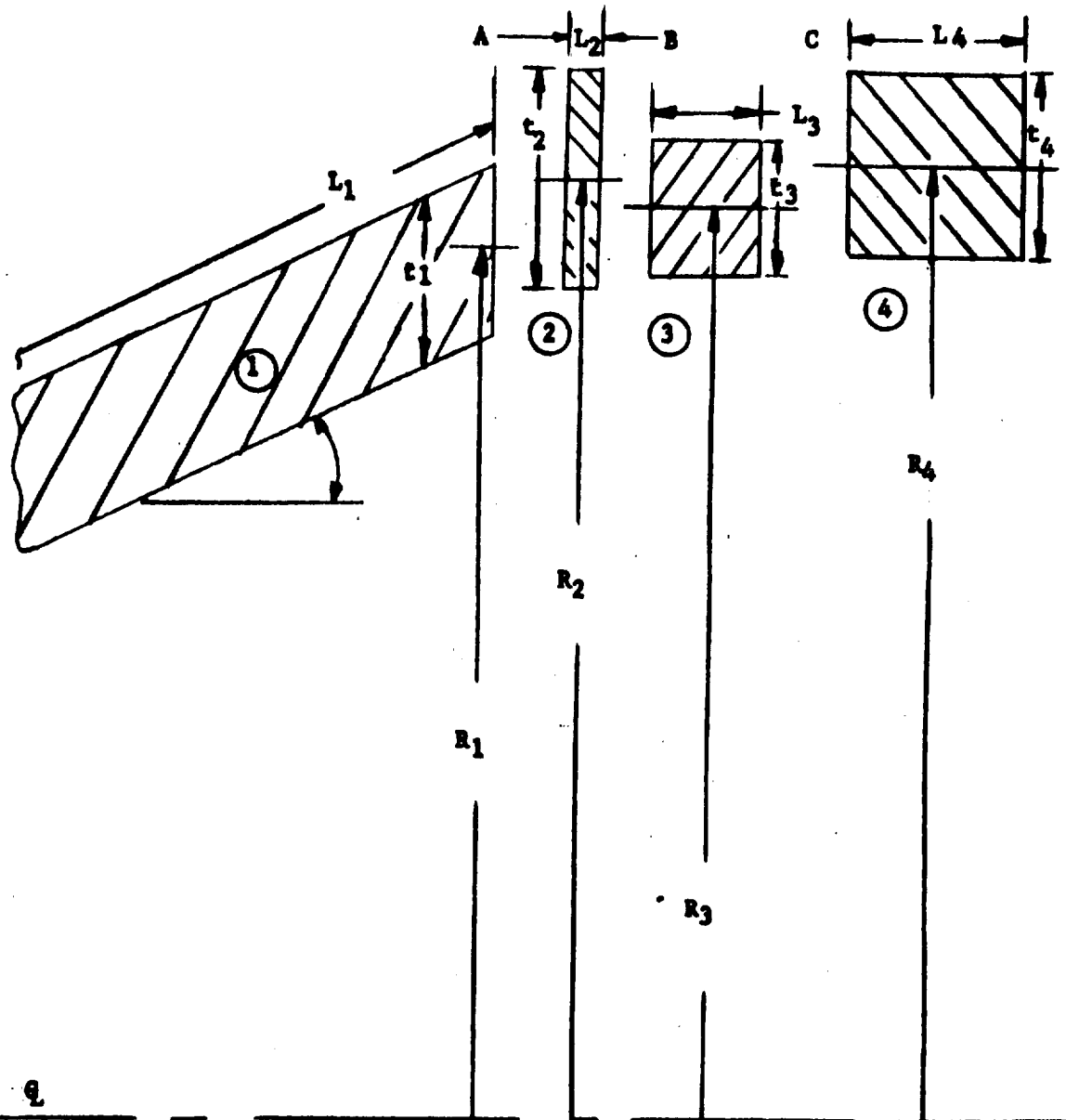
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IDEALIZED BODIES



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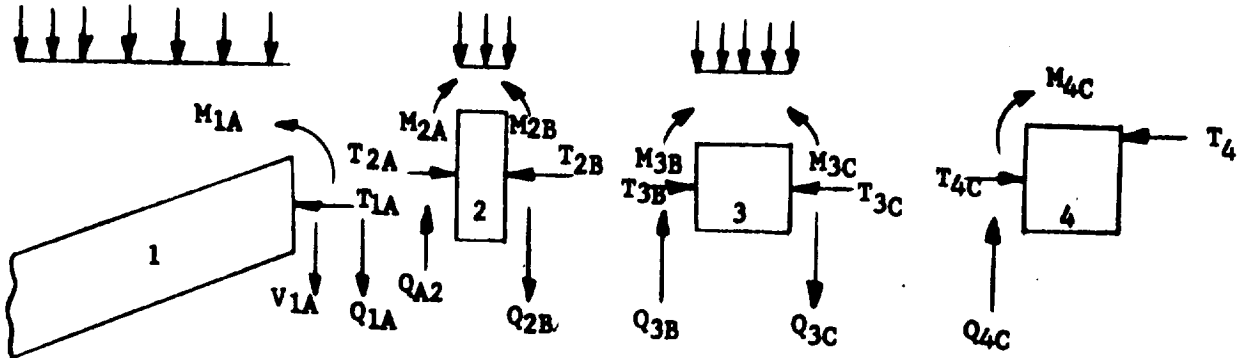
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LOADS ON IDEALIZED BODIES

DIMENSIONS AND EXTERNAL LOADS

BODY 1	BODY 3
$R_1 = 7.20 \text{ in}$ $t_1 = .50 \text{ in}$ $L_1 = 7.0 \text{ in}$ $T_{1A} = 3600 \text{ lb/in}$ $V_{1A} = 1310 \text{ lb/in}$ $P_1 = 1000 \text{ psi}$	$R_3 = 7.30 \text{ in}$ $t_3 = .70 \text{ in}$ $L_3 = .20 \text{ in}$ $T_{3B} = 3650 \text{ lb/in}$ $T_{3C} = 3650 \text{ lb/in}$ $P_3 = 1000 \text{ psi}$
BODY 2	BODY 4
$R_2 = 7.43 \text{ in.}$ $t_2 = .80 \text{ in.}$ $L_2 = .05 \text{ in.}$ $T_{2A} = 3715 \text{ lb/in.}$ $T_{2B} = 3715 \text{ lb/in.}$ $P_2 = 1000 \text{ psi}$	$R_4 = 7.50 \text{ in.}$ $t_4 = .70 \text{ in.}$ $L_4 = .23 \text{ in.}$ $T_{4C} = 3560 \text{ lb/in.}$ $T_4 = 3420 \text{ lb/in.}$ $P_4 = 0 \text{ psi}$



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The influence coefficients, the linear matrix resulting from the discontinuity theory, the internal moments and shear forces, and the stress at each joint were calculated on an electronic computer. Only the necessary results are included.

Young's Modulus (E) = 1.87×10^6

Poissons Ratio (ν) = .25

Linear Matrix

Q _A	M _A	Q _B	M _B	Q _C	M _C	K
.00303	.0885	-.00148	-.0886	0	0	32.786
.0885	.0354	.0886	-.0354	0	0	3116.1
.00148	.0886	.00377	-.0825	.00041	-.00611	32.905
-.0886	-.0354	-.0825	.0360	.00611	-.0611	-.1316.1
0	0	.00041	.00611	.00156	-.00123	4.132
0	0	-.00611	-.0611	-.00123	.1034	36.237

INTERNAL MOMENTS AND SHEAR FORCES

Q _A = -316.4	Q _B = -285	Q _C = -217.5
M _A = 1315	M _B = 928.7	M _C = 879



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DEFLECTIONS AND ROTATIONS

BODY	DEFLECTION LEFT SIDE	DEFLECTION RIGHT SIDE	ROTATION LEFT SIDE	ROTATION RIGHT SIDE
1	.0485	.05577	0	.04142
2	.05577	.05369	.04142	.041428
3	.05369	.04541	.041428	.041428
4	.04541	.03588	.041428	.041428

MERIDIONAL STRESS (S_1) IN PSI.

BODY	S_1 (INSIDE) LEFT SIDE	S_1 (OUTSIDE) LEFT SIDE	S_1 (INSIDE) RIGHT SIDE	S_1 (OUTSIDE) RIGHT SIDE
1	0	0	-324	-8965
2	-324	-8965	-465	-8823
3	6158	-16,590	5549	-15,980
4	5678	-15850	5584	-15360

MINIMUM MARGIN OF SAFETY:

Tension. $M.S. = \frac{6850}{6158} - 1 = .11$

Compression $M.S. = \frac{26,000}{16,590} - 1 = .57$



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HOOP STRESS (s_2) IN. PSI.

BODY	s_2 (INSIDE) LEFT SIDE	s_2 (OUTSIDE) LEFT SIDE	s_2 (INSIDE) RIGHT SIDE	s_2 (OUTSIDE) RIGHT SIDE
1	-15,350	-15,350	-14,120	-16,280
2	-14,120	-16,280	-13,630	-15,720
3	-12,220	-17,900	-10,250	-15,630
4	-8,630	-14,010	-7550	-12,790

MINIMUM MARGIN OF SAFETY:

Compression $M . S = \frac{26,000}{17,900} = 1 = .45$



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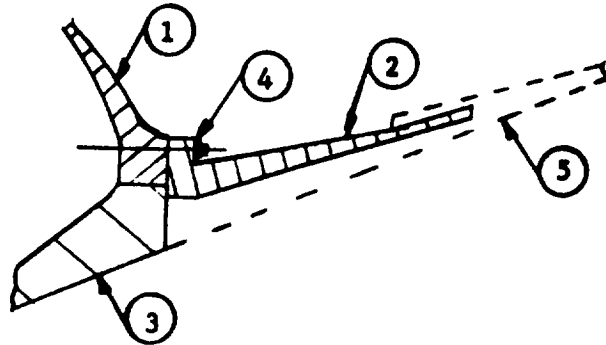
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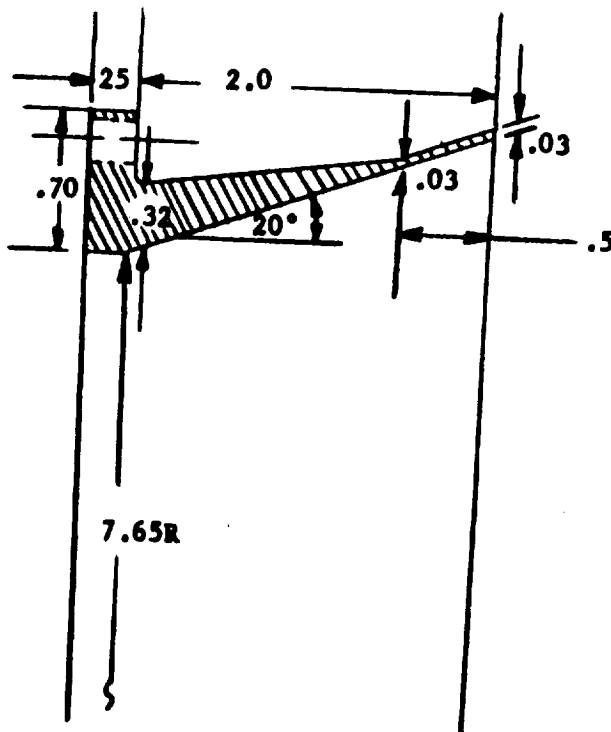
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RETAINING FLANGE AND CONE.



- 1 - MOTOR CASE
- 2 - RETAINING FLANGE AND CONE
- 3 - INTERNAL CONE
- 4 - STUD AND NUT
- 5 - EXTERNAL CONE

ACTUAL DIMENSIONS



MATERIAL:

Titanium

Yield = 125,000 PSI



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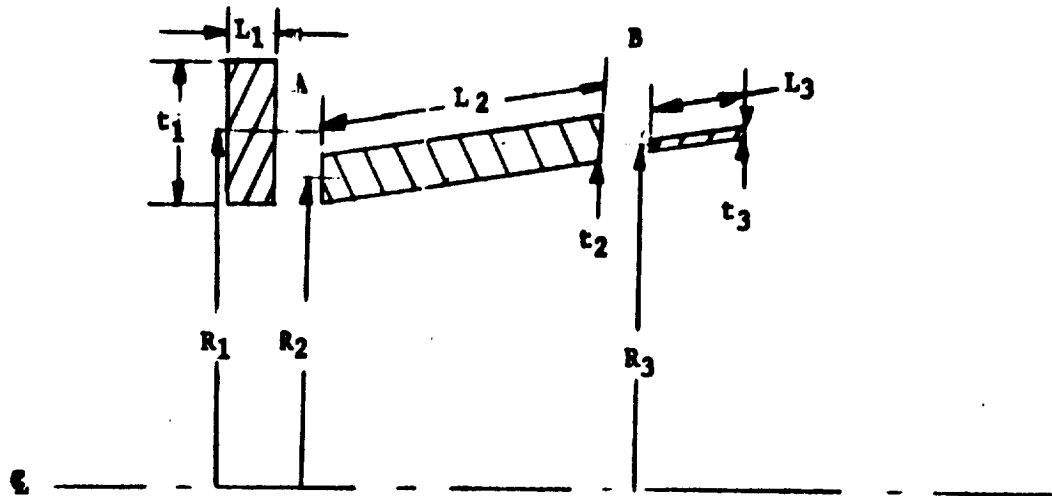
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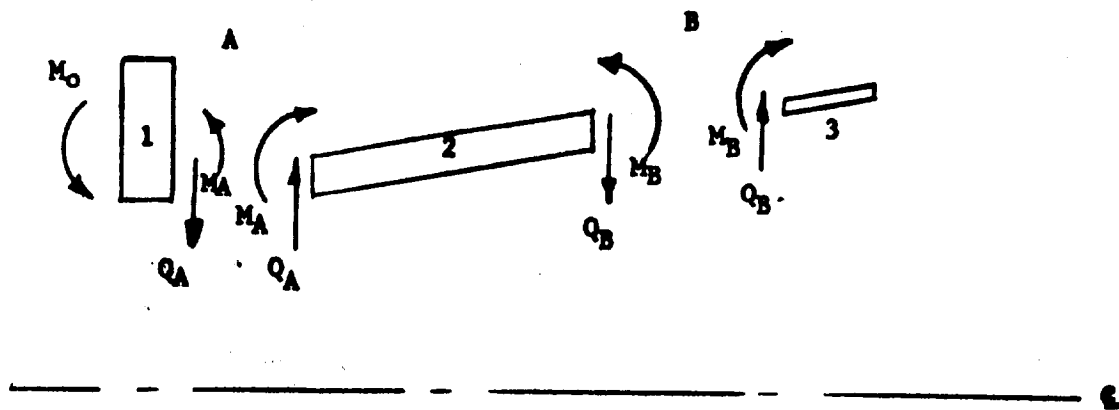
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IDEALIZED BODIES



LOADS ON IDEALIZED BODIES.

BODY 1	BODY 2	BODY 3
$R_1 = 7.98$	$R_2 = 7.75$	$R_3 = 8.65$
$L_1 = .25$	$L_2 = 1.5$	$L_3 = .50$
$t_1 = .70$	$t_2 = .175$	$t_3 = .03$



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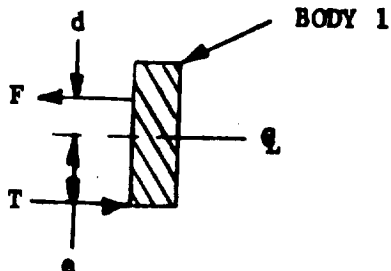
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$$M_o = F \cdot d + T \cdot e$$

$$T = \frac{PR}{2} = \frac{1000 \times 7.83}{2} = 3915 \text{ lb/in}$$

$$F = 3915 \times \frac{7.83}{8.14} = 3775 \text{ lb/in}$$

$$d = .0625 \quad e = .342$$

$$M_o = 3775 \times .0625 + 3915 \times .342$$

$$M_o = 604 + 1337$$

$$M_o = 1941 \text{ in lb/in}$$

The joints are analyzed using discontinuity theory. The influence coefficients, linear matrix, unknown moments and shear forces, and stress were computed on the electronic computer. Only the necessary results are here presented.

$$\text{Young's Modulus} = 16 \times 10^6 \text{ PSI}$$

$$\text{Poisson's Ratio} = .32$$



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LINEAR MATRIX

Q _A	M _A	Q _B	M _B	K
.000152	-.0004744	.00002576	-.00004902	1.0595
-.0004744	.004511	.00004902	-.00005378	-8.4757
.00002576	.00004902	.001337	.003992	0
-.00004902	-.00005378	.003992	.01969	0

INTERNAL MOMENTS AND SHEAR FORCES.

Q _A = 1610.2	Q _B = 85.25
M _A = -1710.7	M _B = -17.94

DEFLECTION AND ROTATION.

BODY	DEFLECTION LEFT SIDE	DEFLECTION RIGHT SIDE	ROTATION LEFT SIDE	ROTATION RIGHT SIDE
1	-.0525	-.02077	.1268	.1268
2	-.02077	+.0359	.1268	.00438
3	+.0359	+.0124	.00438	-.0624

Deflection in inches

Rotation in radians

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MERIDIONAL STRESS (S_1)

BODY	S_1 (INSIDE) LEFT SIDE	S_1 (OUTSIDE) LEFT SIDE	S_1 (INSIDE) RIGHT SIDE	S_1 (OUTSIDE) RIGHT SIDE
1	-23,767	23,767	-20,947	20,947
2	-100,500	100,500	-119,000	119,000
3	-119,000	119,000	0	0

HOOP STRESS (S_2)

BODY	S_2 (INSIDE) LEFT SIDE	S_2 (OUTSIDE) LEFT SIDE	S_2 (INSIDE) RIGHT SIDE	S_2 (OUTSIDE) RIGHT SIDE
1	-112,805	-97,594	-48,350	-34,945
2	-105,140	64,366	28,140	104,686
3	28,140	104,686	23,007	23,007

$$M . S = \frac{125,000}{119,000} - 1 = .05$$



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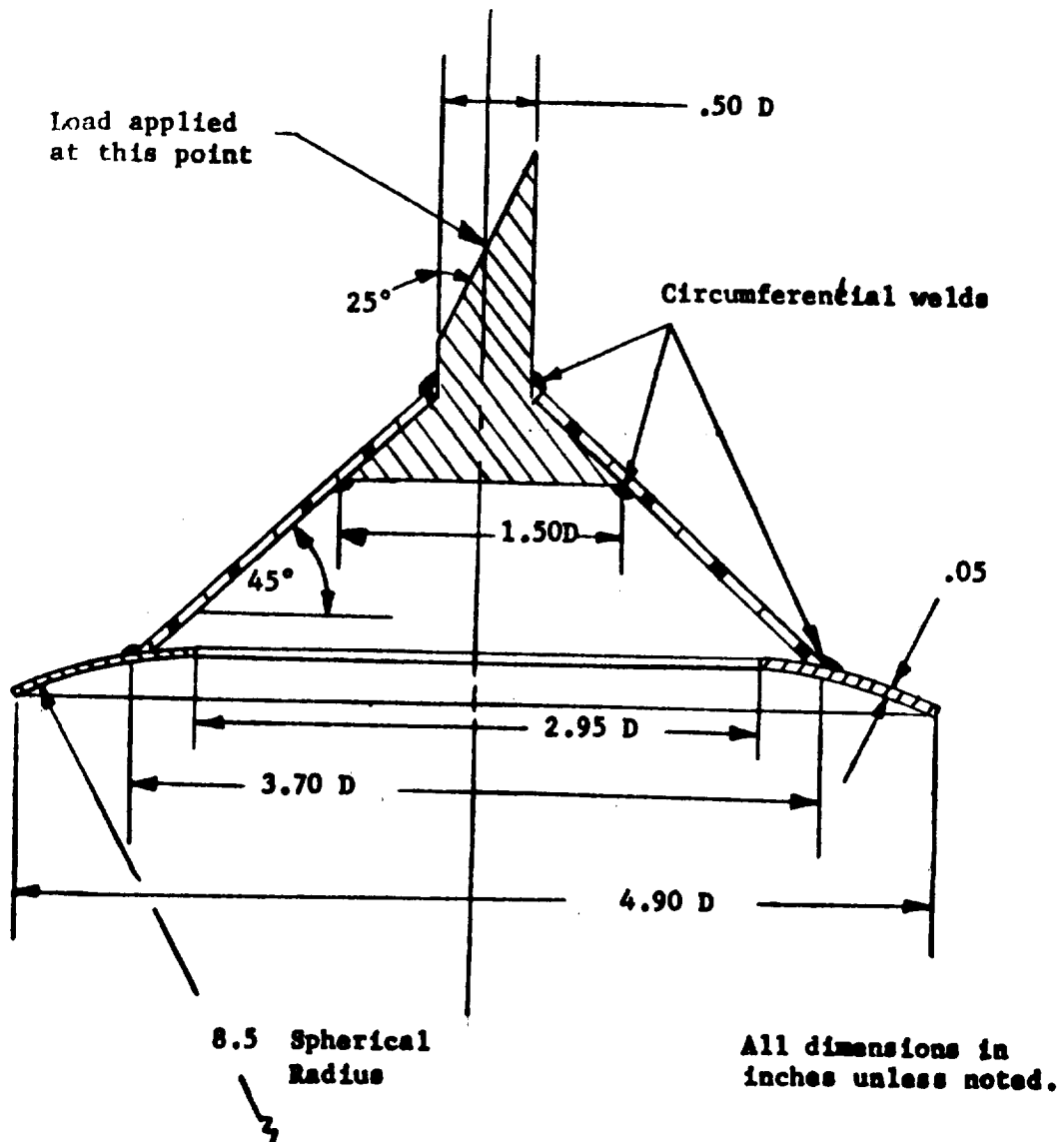
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ATTACHMENT LUGS

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SUBSCALE TEST

One lug was tested to give stress data for full scale design. Specifically, the stress caused in the motor case by the loaded lug was sought. Design requirements call for this case stress to be less than 5000 psi. The test lugs are of the following dimensions:





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ATTACHMENT LUGS

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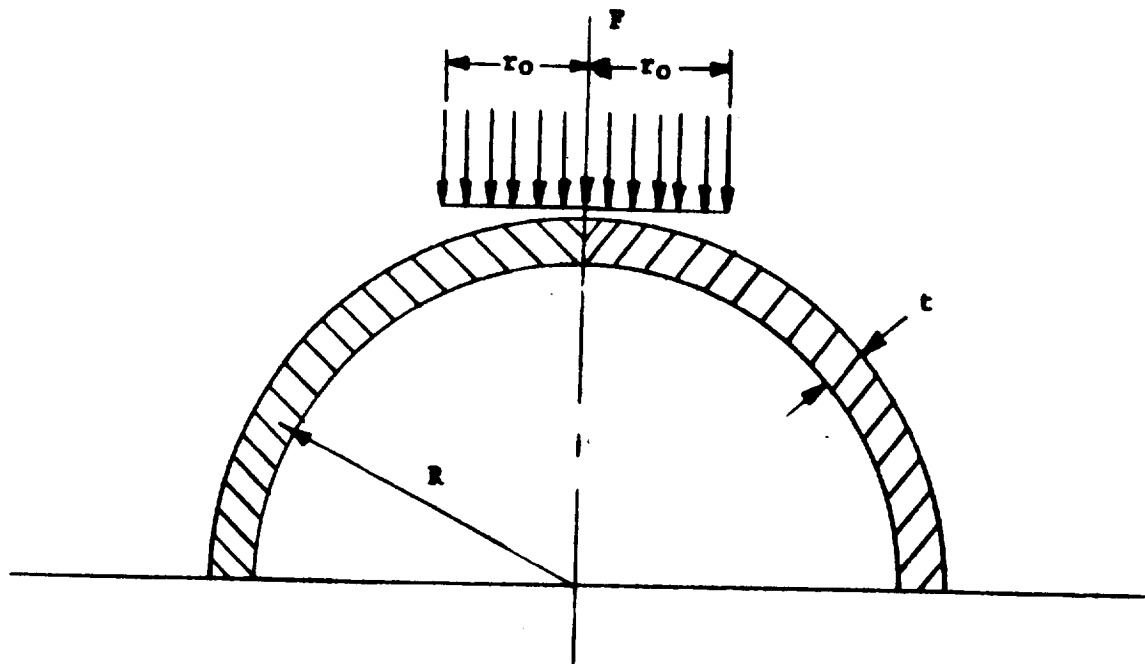
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SUBSCALE TEST

The subscale (test) load was taken as 1/4 of the full scale load. This ratio was taken from the following loading condition on a sphere.*



F = total Force

Membrane Stress:

$$s_1 = s_2 = B \frac{F}{t^2}$$

Bending Stress:

$$s_1' = s_2' = C \frac{F}{t^2}$$

* Reference: Roark Page 273



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SUBSCALE TEST

B and C are nearly linearly dependent on the parameter Br_o

$$Br_o = \frac{1.285 r_o \sqrt{2}}{\sqrt{Rt}}$$

The purpose of the test is to simulate actual stress conditions in the case, therefore the case stress in the subscale should equal the case stress in the full scale. If B and C are kept equal for both the subscale and the full scale, then the Stresses will vary only in the proportion of the term $\sqrt{F/t^2}$. To keep the B and C of the two cases equal, the Br_o of the subscale (Br_{os}) has to equal the Br_o of the full scale (Br_{op}).

The subscript s stands for subscale.

The subscript p stands for full scale.

$$\frac{Br_{op}}{Br_{os}} = \frac{r_{op}}{\sqrt{R_{ptp}}} \times \frac{\sqrt{R_{ats}}}{r_{os}} = 1$$

$$r_{op} = r_{os} \left[\frac{R_{ptp}}{R_{ats}} \right]^{1/2}$$

$$r_{op} = r_{os} \left[\frac{36 \times .06}{17 \times .03} \right]^{1/2}$$



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SUBSCALE TEST

$$\therefore r_{op} = 2 r_{os}$$

$$\frac{F_s}{F_p} = \frac{t_s^2}{t_p^2} = \left(\frac{.03}{.06} \right)^2 = 1/4$$

$$F_s = .25 F_p$$

FULL SCALE LOAD

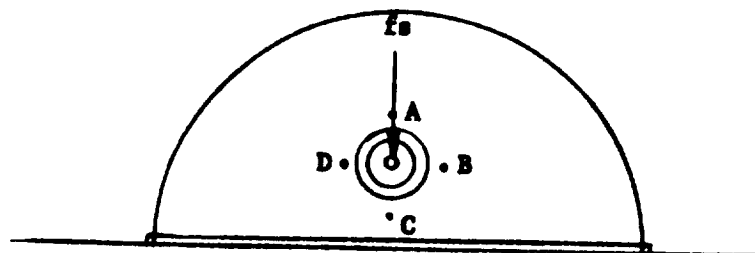
$$F_p \text{ (Total)} = \frac{1500\#}{8} \times 10g = 15,000\#$$

$$f_p = \frac{15,000\#}{3 \text{ Lugs}} = 5000 \#/\text{Lug}$$

SUBSCALE LOAD

$$F_s = (.25) (5000) = 1250 \#/\text{Lug}.$$

SUBSCALE TEST RESULTS



All gauges
parallel to
load line.

Location of strain gauges

Gauge B burned out during balancing.



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SUBSCALE TEST RESULTS

GAUGE	WALL THICKNESS
A	.033 inches
B	.032
C	.0325
D	.032

$$\sigma = \frac{E}{1 - \nu} E_M$$

 σ = Stress

E = Modulus of Elasticity

 ν = Poisson's Ratio E_M = Measured Strain

$$\frac{E}{1 - \nu} = 25.715 \times 10^6 \text{ psi}$$

This value was determined in the static pressure test of a subscale (Titanium) motor.

STRESS

LOAD	GAUGE A	GAUGE C	GAUGE D
250 #	1930 psi	1285 psi	1030 psi
500	2940	1930	1805
750	5140	900	2440
1000	7070	-1030	2940
1250	6030	-2940	3990
1365	CORE FAILED		



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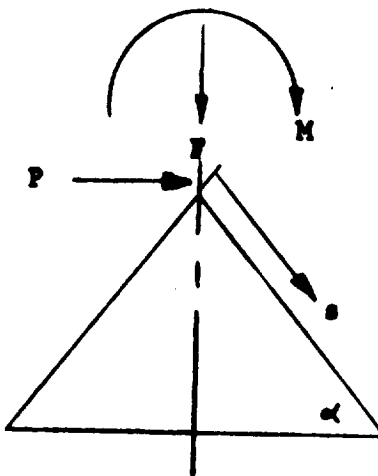
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ANALYSIS OF CONICAL LUG.*

(Subscale)



$$N_s = \frac{C}{s} + \frac{B}{s} + \frac{n}{\cos} \frac{A}{s^2}$$

$$N_{s\theta} \text{ (Hoop)} = \frac{A}{s^2}$$

$$N_{\theta} = 0$$

$$M = A \pi \cos \alpha \cdot \sin \alpha$$

$$P = B \pi \cos^2 \alpha$$

$$F = C \pi \sin 2\alpha$$

* Reference: Flügge Page 66

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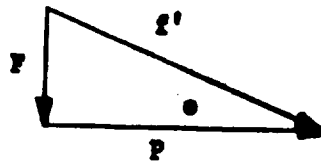
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ANALYSIS (Continued)

(Subscale)



$$f' = 1250$$

$$\theta = 25^\circ$$

$$P = f' \cos \theta = 1132\#$$

$$F = f' \sin \theta = 527\#$$

$$M = 1132 \times .5 = 566 \text{ in}\#$$

$$A = \frac{566}{3.14 \times .707 \times .707} = 361$$

$$B = \frac{527}{3.14 \times 1.0} = 168$$

$$N_s = \frac{168}{s} + \frac{724}{s} + \frac{1}{.707} \frac{361}{s^2}$$

$$N_{s\theta} = \frac{892}{s} + \frac{398}{s^2}$$

A2



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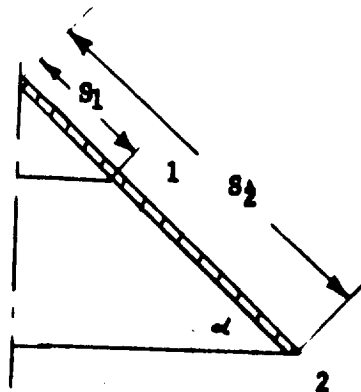
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ANALYSIS (Continued) (Subscale)



$$\alpha = 25^\circ$$

$$s_1 = .826$$

$$s_2 = 2.20$$

Point 1 is at weld of cone to end.

Point 2 is at weld of cone to base.

At 1

$$N_s = \frac{892}{.826} + \frac{398}{.826} = 1662$$

$$N_{s\theta} = \frac{361}{.826} = 529$$

At 2

$$N_s = \frac{892}{2.2} + \frac{398}{2.2} = 488$$

$$N_{s\theta} = \frac{361}{2.2} = 75$$



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ANALYSIS (Continued)

(Subscale)

Stress at 1

$$\sigma_s = \frac{N_s}{t} = \frac{1662}{.05} = 33,100 \text{ PSI}$$

$$\sigma_{s\theta} = \frac{N_{s\theta}}{t} = \frac{529}{.05} = 10,600 \text{ PSI}$$

Stress at 2

$$\sigma_s = \frac{488}{.05} = 9760 \text{ PSI}$$

$$\sigma_{s\theta} = \frac{75}{.05} = 1500 \text{ PSI}$$

Yield Stress of Aluminum (615-T6) used is 36,000 PSI*

If f' is increased to 1365 (Failure)

$$\sigma_s = 36,100 \text{ PSI}$$

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DESIGN OF FULL SCALE LUGS.

$$f = 5000\#$$

$$P = 5000 \times .907 = 4530\#$$

$$F = 5000 \times .423 = 2110\#$$

$$M = 4530 \times .2 = 905 \text{ in}\#$$

$$A = \frac{905}{3.14 \times .707 \times .707} = 576$$

$$B = \frac{4530}{3.14 \times .707^2} = 2760$$

$$C = \frac{2110}{3.14 \times 1.0} = 672$$

$$N_s = \frac{672}{s} + \frac{2760}{s} + \frac{1}{.707} \times \frac{576}{s^2}$$

$$N_{s0} = \frac{576}{s^2}$$

$$N_s = \frac{3432}{s} + \frac{815}{s^2}$$

Using Titanium and a maximum stress of 150,000 psi, the wall thickness of the conical lug should decrease from the top to the base.



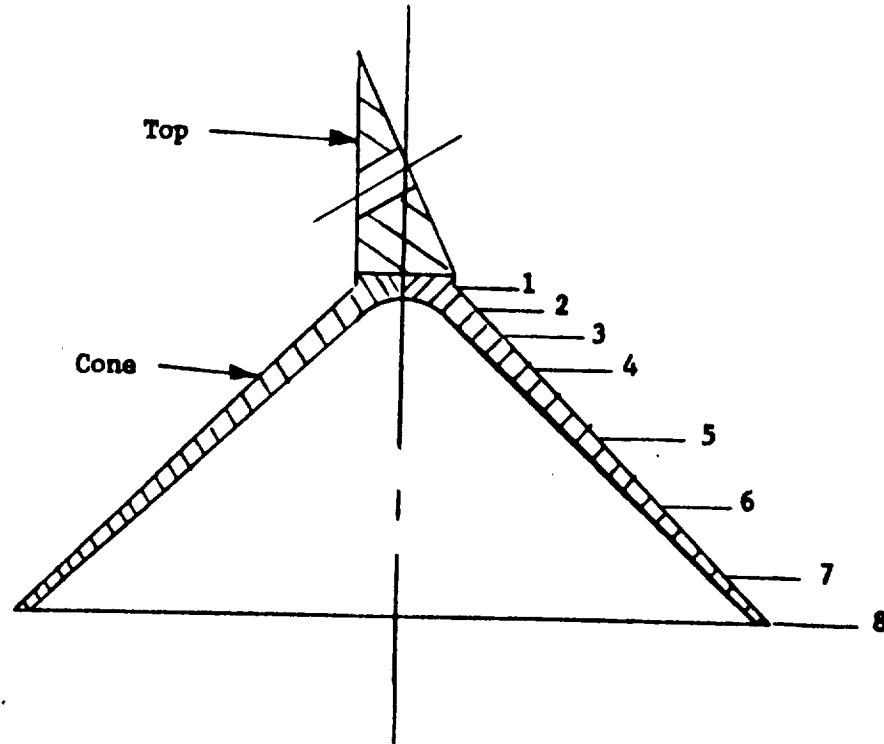
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DESIGN (Continued)



STATION	S	Ns	Ns 0	t*
1	.71	6465	1150	.043 in
2	1.0	4247	576	.029
3	1.5	2647	256	.018
4	2.0	1918	144	.013
5	3.0	1232	64	.009
6	4.0	910	36	.007
7	5.0	729	23	.005
8	5.65	632	18	.0045

$$t = \frac{N_s}{\sigma} \frac{N_s}{150,000}$$



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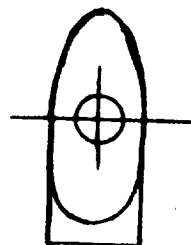
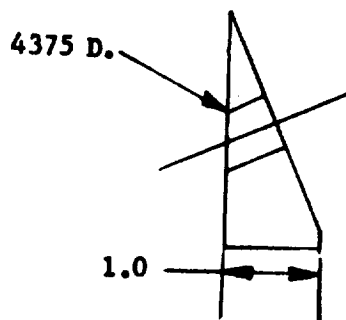
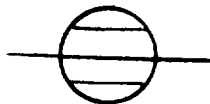
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USING SIMPLE BEAM THEORY

$$\sigma = \frac{Mc}{I} \quad (\text{at Point A})$$

$$M = F \times L = 5000 \times .580 = 2880$$

$$I = \frac{\pi r^4}{4}$$

$$c = r$$

$$\sigma = \frac{2880 \times .5 \times 4}{3.14 \times (.5)^3} = 29,400 \text{ PSI}$$

$$M.S. = \frac{150,000}{29,400} - 1 = 4.1$$



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JPL MOTOR

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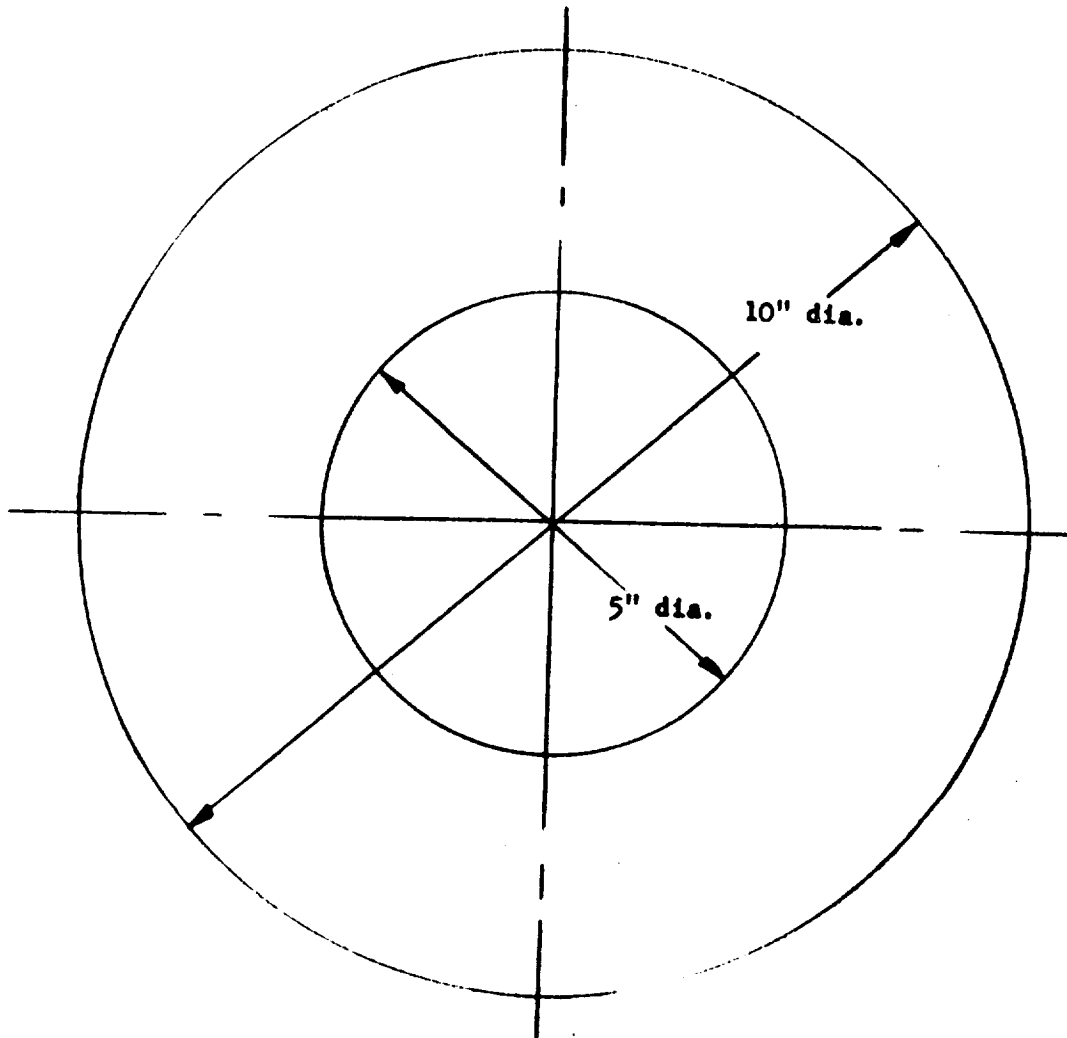
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Design (continued)

The original design criteria called for $rop = 2 \text{ ros}$. Since stresses were 1.4 too high the base area should be increased. An increase in base area by a factor of 1.4 would increase the rop by 1.19. Therefore $rop = 2.38 \text{ ros}$. The base dimensions would be the following.





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Johns, R.H. and Orange, T.W.; "Theoretical Elastic Stress Distributions Arising From Discontinuities and Edge Loads In Several Shell-Type Structures". NASA TR R-103, 1961

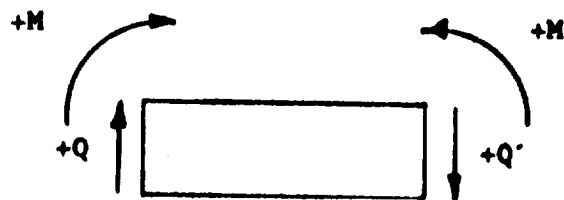
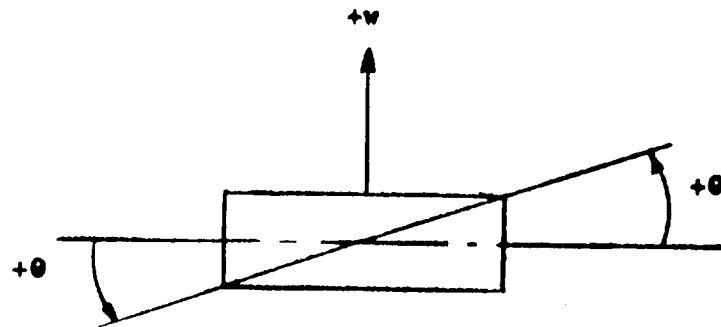


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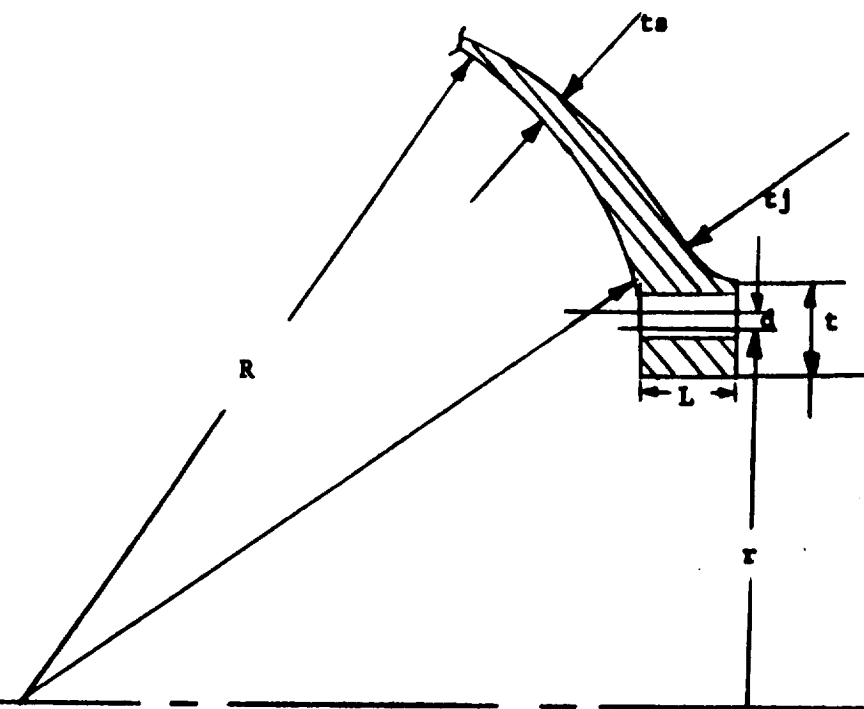
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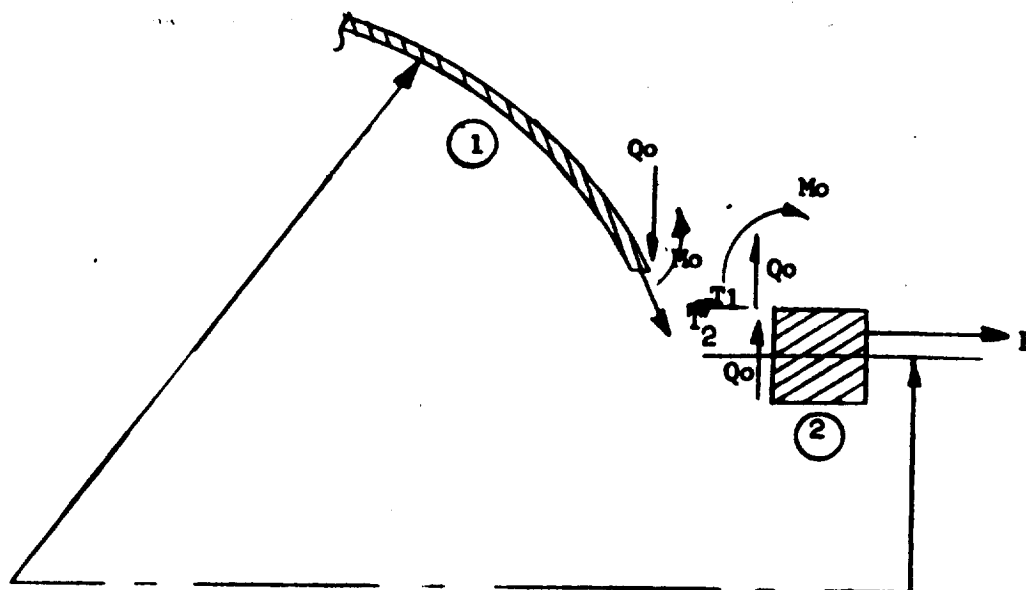


$R = 8.5$
 $t_s = .03$
 $t = .4375$
 $L = .312$
 $r = 4.219$
 $d = .0625$
 $t_j = .111$

REF, ARC

DWG No.

SK 5-23861



Loads On Idealized Bodies

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SPHERICAL INFLUENCE COEFFICIENTS

$$\begin{aligned} R &= 8.5 \\ t &= .03 \\ \alpha &= 31.4 \\ \Phi &= 180 - 31.4 = 148.6 \\ \sin \phi &= .521 \\ \cos \phi &= .854 \\ \cot \phi &= -1.658 \end{aligned}$$

$$\lambda = 1285 \sqrt{\frac{R}{t}} = 21.63$$

$$K_1 = 1 + \frac{(1 - 2\nu) \cot \phi}{2 \lambda}$$

$$K_1 = 1.0152$$

$$K_2 = 1 + \frac{(1 + 2\nu) \cot \phi}{2 \lambda}$$

$$K_2 = 1.0609$$

$$\delta_{1Q} = \frac{21.63 \times 8.5 \times .521^2 \times 1.957}{E \times .03} = \frac{3255}{E}$$

$$\delta_{1M} = \phi_{1Q} = \frac{2 \times 21.63^2 \times .521}{E \times .03 \times 1.0152} = \frac{16,000}{E}$$

$$\phi_{1M} = \frac{4 \times 21.63^3}{E \times .03 \times 8.5 \times 1.0152} = \frac{156,360}{E}$$

$$W_{1P} = \frac{.35 \times 1000 \times 8.5^2 \times .521}{E \times .03} = \frac{43,914}{E}$$

$$T = \frac{PR}{2} = \frac{1000 \times 8.5}{2} = 4250$$

$$T_2 = T \sin \alpha = 4250 \times .521 = 2214$$

$$T_1 = T \cos \alpha = 4250 \times .854 = 3630$$

$$aT_2 = .2188 \times 2214 = 490$$



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SHORT CYLINDER COEFFICIENTS

$$r = 4.219 \quad B = \frac{1.285}{\sqrt{4.219 \times .4375}} = .946$$

$$t = .4375$$

$$L = .312 \quad BL = .946 \times .312 = .295$$

$$\frac{1}{D} = \frac{10.92}{Et^3} = \frac{10.92}{E \times .4375^3} = \frac{130.4}{E}$$

$$\delta_{2Q} = \frac{1}{.295} \times \frac{130.4}{(.946)^3 E} = \frac{522}{E}$$

$$\delta_{2M} = \phi_{2Q} = \frac{1.5}{(.295)^2} \times \frac{130.4}{(.946)^2 E} = \frac{2512}{E}$$

$$\phi_{2M} = \frac{3}{(.295)^3} \times \frac{130.4}{.946 E} = \frac{16,000}{E}$$

$$w_{2p} = \frac{.85 \times 1000 \times (4.219)^2}{E \times .4375} = \frac{34,580}{E}$$

$$F = \frac{PR_1^2}{2R_2} = \frac{1000 \times 4^2}{2 \times 4.25} = 1882$$

$$F \cdot d = 1882 \times .0625 = 117.6$$



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USING EQUATIONS DERIVED FOR 36 INCH CASE

$$(\delta_{1Q} + \delta_{2Q}) Q_0 + (\delta_{2M} - \delta_{1M}) M_0 = W_{1P} - W_{2P} + \delta_{2M} (aT_2 - F \cdot d) - \delta_{2Q} T_1$$

$$(\delta_{2Q} - \delta_{1Q}) Q_0 + (\delta_{1M} + \delta_{2M}) M_0 = \delta_{2M} (aT_2 - F \cdot d) - \delta_{2Q} T_1$$

$$3777 Q_0 - 13,488 M_0 = 551,740$$

$$-13,488 Q_0 + 172,460 M_0 = 5,322,000$$

$$Q_0 - 3.571 M_0 = 146$$

$$-Q_0 + 12.786 M_0 = 395$$

$$9.215 M_0 = 541$$

$$M_0 = 58.7$$

$$Q_0 = 356$$

$$\frac{PR}{2t} + \frac{6 M_0}{t^2} = 147,000$$

$$\frac{4250}{t} + \frac{352}{t^2} = 147,000$$

$$147 t^2 + 4.25 t + .352 = 0$$

$$t = .07 \text{ in}$$



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$$\frac{6 M}{t^2} = 5000 \text{ at } t = .03$$

$$M_w = .75$$

$$M_0 \cdot e^{-\lambda w} = M \text{ (Design formula)}$$

$$\exp(-\lambda w) = \frac{.75}{58.7} = .01297$$

$$\lambda w = 4.35$$

$$w = \frac{4.35}{21.63} = .201 \text{ RAD}$$

$$w = .201 \times 57.35 = 11.5^\circ$$

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APPENDIX E

INDUSTRIAL HYGIENE AND AIR POLLUTION CONTROL

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INDUSTRIAL HYGIENE AND AIR POLLUTION CONTROL

It was thought that a review of the complete industrial-hygiene and air-pollution control program in effect at that part of the Pine Ridge Experiment Station devoted to work with beryllium would be more meaningful than coverage of only the portion related directly to the work of this contract. Accordingly, this section describes and gives the results of the entire program.

PROGRAMS AND PROCEDURES

Medical Standards and Controls

Physical fitness requirements and medical surveillance standards adopted for the protection of personnel assigned to this program were described previously.¹ A scheduled annual re-examination and X-ray checks show no evidence of health injury related to beryllium exposure, in any project employee. In addition to the medical history records,² a weekly weight record for each project employee has been instituted.

Industrial Hygiene Standards

The industrial hygiene standards adhered to during this project are those promulgated by the Advisory Committee of the United States Atomic Energy Commission in 1949. They provide that: (1) in-plant atmospheric concentrations

¹ Atlantic Research Corporation, Annual Technical Summary Report, July 1, 1959 through June 30, 1960. Contract AF 33(616)-6623, Project No. 3059, Task No. 30312, ARPA Order No. 24-59, Task 4, September 1960. CONFIDENTIAL.

² Atlantic Research Corporation, Annual Technical Summary Report, July 1, 1960 through June 30, 1961. Contract AF 33(616)-6623, Project No. 3059, Task No. 30312, ARPA Order No. 24-60, Task 4, September 1961. CONFIDENTIAL.

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of beryllium should not exceed $2 \mu\text{gm}/\text{m}^3$ as an average concentration throughout an 8-hour day; (2) even though the daily average exposure is no more than $2 \mu\text{gm}/\text{m}^3$, no personnel should be exposed to a concentration greater than $25 \mu\text{gm}/\text{m}^3$ for any period of time, however short; and (3) in the neighborhood of a plant handling beryllium compounds, the average monthly concentrations should not exceed $0.01 \mu\text{gm}/\text{m}^3$.

Air-Pollution Control Devices

Air-pollution control devices were the same as those already described.¹

Uniforms and Safety Equipment

Uniforms and safety equipment during this contract were essentially the same as those used previously. However, the Wilson #809 Respirator with R520 super filters supplied by the Wilson Product Division of Ray-O-Vac Company, Reading, Pennsylvania, was adopted for routine respiratory protection. The face piece of this mask is made of softer rubber than that in the American Optical respirator previously used and personnel found it both more comfortable and to provide better fit over a wider range of facial contours than the masks it replaced. Furthermore, with this respirator, the wearer may quickly check for the adequacy of face-piece fit by occluding the inlet holes to the respirator cartridge, a maneuver not possible with the American Optical respirator.

Beryllium-Contaminated Trash Disposal

An incinerator for burning beryllium-contaminated trash and waste beryllium propellant was installed in the large firing chamber. The incinerator consists of a steel trough 30 inches wide by 5 feet long with a

¹ Ibid.

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kerosene-fueled blast burner (Blackwell TD-10-X5) mounted on the upstream end of the trough to (1) evaporate the water with which some batches of trash are inserted and (2) ignite the wastes. The incinerator is fed remotely through a chute located in the side of the firing tunnel. When operating with water-saturated wastes, the capacity of the incinerator is approximately one 30-gallon drum of waste per hour. During operation, the combustion air enters the firing chamber through the thrust-stand end of the chamber and is exhausted through the Pease-Anthony Scrubber to the atmosphere. The large volume (approximately 7,000 cu ft) within the chamber is sufficient to absorb any surges in gas evolution as a result of detonation of chunks of waste propellant or ignition of solvent vapors without the exhaust products surging out through the air inlet.

Monitoring Program

Air and gas samples were collected routinely to evaluate process operation, industrial-hygiene hazards, and air-pollution control efficiency. The system used for classification of these samples has been reported previously¹ and is followed in the report of analytical results in this section. Within each class of sample, the sampling rate was maintained uniform from sample to sample so that a volume weighting of a number of samples also results in a time-weighted average.

The high-volume samplers previously used as perimeter samplers were replaced by a positive-displacement sampling train. This sampling train consists of a laboratory-designed filter support fitted with an 11-cm Whatman No. 41 filter paper, a positive-displacement Sprague Model 240 dry gas meter equipped with suction gauge and thermometer, and a 5-cfm Bell and Gossett oil-less vacuum pump. A running-time meter in parallel with the vacuum-pump motor permits recognition of any interruption in power supply to the device and the

¹Ibid.

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duration of such interruption. These samplers have proved to be much more reliable, and precise for sample volume indication, and maintenance problems have been greatly decreased as compared with the samplers previously used.

Surface wipe samples were collected routinely through the early part of the program (1) to evaluate the adequacy of clean-up and housekeeping operations in areas where beryllium was handled and (2) to detect and quantify any dissemination of beryllium by foot traffic and/or dust fall. Samples were collected by scrubbing a measured area of the surface under investigation with filter paper or cellulose wipes wet with distilled water. Surface wipe sampling, however, was discontinued as the experience of the technical personnel increased and regularly scheduled clean-up of all areas was considered adequate.

The results of the industrial-hygiene monitoring program are shown in Tables I and II.

It is to be stressed that the values presented in these tables are not to be considered average values for the site. To economize on sampling and analytical time and cost without sacrificing sensitivity in our ability to detect deteriorating or unsatisfactory operating conditions, major emphasis was placed on sampling the potentially troublesome areas. Areas which, on the basis of past experience were known to have consistently low levels of beryllium contamination, were sampled only rarely. Many of the high levels reported occurred as a result of a series of trash fires in the hot-trash waste disposal cans in the beryllium propellant laboratory. On each such occasion, a large number of samples were taken during and immediately after the event to provide guidance in decontamination and clean-up and to provide good historical coverage.

Analytical Techniques

The "ZENIA" colorimetric procedure described in the second Atlantic Research Corporation Annual Technical Summary Report,¹ has been replaced by

¹Ibid.

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TABLE I
BERYLLIUM CONCENTRATION OF AIR SAMPLES FOR PERIOD JANUARY 1, 1962 THROUGH SEPTEMBER 30, 1962

Sample Class	January 1962			February 1962			March 1962			April 1962			May 1962		
	Number of Samples	Beryllium Concentration $\mu\text{gm}/\text{cu m}$	Mean	Number of Samples	Beryllium Concentration $\mu\text{gm}/\text{cu m}$	Mean	Number of Samples	Beryllium Concentration $\mu\text{gm}/\text{cu m}$	Mean	Number of Samples	Beryllium Concentration $\mu\text{gm}/\text{cu m}$	Mean	Number of Samples	Beryllium Concentration $\mu\text{gm}/\text{cu m}$	Mean
Perimeter neighborhood	12	0.01	0.05	9	0.001	0.02	8	0.003	0.016	6	0.001	0.004	7	0.001	0.003
Project site outdoors	14	0.05	0.35	10	0.05	0.18	9	0.03	0.23	3	0.01	0.02	2	0.05	0.21
Office areas, clean labs	7	0.18	0.45	8	0.04	2.1	3	0.05	0.11	6	0.04	0.48	1	0.0	0.0
Process areas	33	0.29	1.4	24	0.27	8.7	34	0.37	7.8	46	0.34	4.7	59	0.14	4.2
Stack inlet	3	10.9	3.4	--	--	--	--	--	--	--	--	--	--	--	--
Stack outlet	33	6.0	75.2	27	0.26	2.3	29	4.8	64.7	24	0.24	4.4	16	0.53	1.6

Sample Class	June 1962			July 1962			August 1962			September 1962		
	Number of Samples	Beryllium Concentration $\mu\text{gm}/\text{cu m}$	Mean	Number of Samples	Beryllium Concentration $\mu\text{gm}/\text{cu m}$	Mean	Number of Samples	Beryllium Concentration $\mu\text{gm}/\text{cu m}$	Mean	Number of Samples	Beryllium Concentration $\mu\text{gm}/\text{cu m}$	Mean
Perimeter neighborhood	6	0.001	0.003	12	0.002	0.04	8	0.0000	0.002	7	0.0007	0.005
Project site outdoors	--	--	--	1	0.007	0.007	--	--	--	--	--	--
Office areas, clean labs	--	--	--	8	0.14	0.77	1	0.0	0.0	--	--	--
Process areas	30	0.06	2.6	26	0.19	1.18	33	0.17	2.11	43	0.28	1.73
Stack inlet	--	--	--	--	--	--	--	--	--	--	--	--
Stack outlet	6	0.51	1.7	12	0.50	13.0	23	0.90	23.1	14	0.29	0.97

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a modification of the Owens and Yoe colorimetric method. A number of improvements have been made in adapting this procedure to the requirements of the project. A detailed analytical technique is presented in Appendix F of the third Atlantic Research Corporation Annual Technical Summary Report.¹ Chief problems encountered with this procedure have been pH control, positive interferences from other metals, and cross-contamination from adsorption of beryllium on analytical glassware. The latter problem is still not solved and is temporarily being met by discarding glassware contaminated by high-beryllium-level samples.

Late delivery, mechanical and electronic difficulties, and the unavailability of adequate calibration standards prevented the use of the National Spectrographic Laboratory Beryllium Monitor for routine industrial-hygiene control and monitoring. A more detailed discussion of the difficulties experienced with this instrument and a tentative evaluation of the limitations and capabilities of the instrument are separately presented in Appendix F of the third Atlantic Research Corporation Annual Technical Summary Report.

SPECIAL PROBLEMS

The number and variety of beryllium-using projects being carried out has led to an increase in the number of individuals handling powdered beryllium. As a consequence, the quality of industrial-hygiene discipline has, at times, been lower than is attainable with a crew of long experience. Intensified industrial-hygiene monitoring was used during training periods, and it is under such circumstances that many of the high wipe-sample readings were obtained. In all cases, it was possible to reduce indicated high levels to normal, acceptable values by an intensified effort to tighten housekeeping and clean-up procedures.

¹Atlantic Research Corporation, Annual Technical Summary Report, July 1, 1961, through June 30, 1962, Contract AF 33(616)-6623, Project No. 3059, Task No. 30312, ARPA Order No. 24 Amendment 38, September 1962. ~~CONFIDENTIAL~~

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The replacement of a plastic-film glove box by a rigid-walled glove box with more adequate and easily operated seals and pass-through ports substantially lowered the incidence of high readings in the area of the box.

The trash-can fires previously referred to were apparently initiated by exothermic reaction of waste propellant ingredients from developmental work. In all such instances, personnel involved were either wearing respirators or donned them immediately. Exposures of unprotected personnel to concentrations above the acceptable tolerances were not experienced. In the instance of a spill of beryllium powder, which was the cause of the highest reading of the year for air samples in the process areas, the operator was wearing a respirator. This incident indicates the value of the standard operating procedure of wearing respirators when hand-carrying powdered beryllium, whether in a closed container or not.

The fires and the spill occurred in areas which were easily isolated with no danger of significant contamination to other areas. Samples taken during these occurrences showed a rapid decay in airborne beryllium concentrations, and clean-up in the areas was accomplished quickly. Respiratory protective equipment was used during such clean-up.

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